



ANALYSIS OF RADIALY SYMMETRIC TEXTURES USING PARTIAL POLE FIGURES

INTRODUCTION

Many manufacturing processes, including uniaxial compression, wire drawing, rolling, and chemical vapor deposition (CVD), tend to orient crystals in the material with a specific crystallographic plane parallel to a certain direction (i.e. compression axis, drawing axis, sheet normal, and surface normal). These manufacturing processes produce a preferred orientation in materials known as "texture." The presence and degree of texture can have a profound effect on the material's mechanical, physical, and/or electrical properties.

Textures are described by means of pole figures. A "pole" is the vector normal to a crystal plane. Pole figures are stereographic projection plots of the variation in pole density with orientation in the sample for a certain crystal plane.

Many thin films are deposited with a specific (hkl) plane parallel to the plane of the surface. The pole figure for such a film will be rotationally symmetric and indicate a high concentration of poles oriented normal to the surface with decreasing pole density. This occurs as the angle between the surface normal and crystal orientation increases. If a pole figure can be obtained from the crystal plane, which is preferentially oriented parallel to a sample's surface, a partial pole figure plot can be calculated from the pole figure data as a means of quantifying the degree of texture present.

For each angle of tilt, ϕ (with $\phi=0$ corresponding to a crystal with the (hkl) plane parallel to the surface), the partial pole figure plots the fraction of crystals which are oriented between ϕ and 0 deg. A computer program has been written, based on the method of measuring partial pole figures

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ANNOUNCEMENTS

Laser Shocking Residual Stress Studies

Lambda Research has completed a number of studies of residual stress distributions developed both as a function of depth and distance from the center of shock zones on laser processed components. Techniques and apparatus for high precision sample positioning were developed in order to accurately locate the x-ray beam during measurement. The positioning technology has been used for a variety of high speed resolution residual stress studies including split sleeve cold expanded (SSCE) holes. Contact: Perry Mason

Residual Stress and Welded Alloy 600 Heater Sleeves

An EPRI funded study of the residual stress distributions developed in alloy 600 heater sleeves welded into pressure vessels at an angle of 50 deg. has been completed in conjunction with ABB-Combustion Engineering Nuclear Power of Windsor, Connecticut. The results of this study provide an excellent example of the effect of prior cold working on residual stresses developed by the complex shrinkage associated with welds. The paper has been published and was recently presented by Jim Molkenhuth of ABB-Combustion Engineering at the Sixth International Symposium on Environmental Degradation of Materials in Nuclear Power Systems-Water Reactors. Copies of the paper are available. Contact: Paul Prevey

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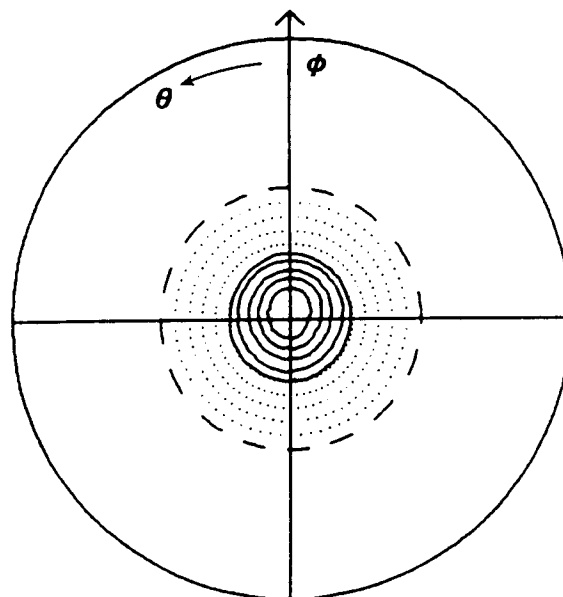


developed by Yang¹, which calculates the partial pole figure from the data obtained for a full pole figure.

APPLICATION

To demonstrate the technique, partial pole figure analysis is performed on a 2μm thick CVD grown ZnO film with a suspected (0002) texture. The purpose of the analysis is, first, to measure the (0002) pole figure to confirm the suspected texture. Then, provided texture in the film has the required radial symmetry, the partial pole figure is calculated to obtain a quantitative measure of the degree of texture.

In measuring the (0002) pole figure, a diffraction pattern, shown in Figure 1, is obtained to determine the positions of the ZnO (0002) peak and any peaks from the substrate. The (0002) pole figure of the ZnO film sample is obtained on a stepper-motor driven Schulz back-reflection pole figure device mounted on a Bragg-Brentano focusing geometry horizontal diffractometer using copper K-alpha radiation. Although the film is transparent to the copper K-alpha radiation used, there is no overlap between the diffraction peaks of the substrate and the ZnO film, which would interfere with the measurement of the (0002) pole figure.



(0002) Stereographic Projection
Contours at 1 2 4 6 8 X Random

Fig. 2 - (0002) Pole Figure of the ZnO Coating

The diffracted intensity of the (0002) peak is collected for one second intervals at increments of 4 deg. on a nominally equal area spherical net throughout the central 50 deg. of the pole figure. The background intensity is measured on either side of the diffraction peak at each angle of tilt and interpolated to determine the background component of the diffraction peak. Since a randomly oriented ZnO sample is not available, corrections for defocusing are made with reference to the (1011) peak of a titanium random powder sample (chosen for its similar 2-theta position and peak width as compared to the ZnO (0002) peak). Corrections are also made for intensity losses due to the transparency of the film to copper radiation. After subtraction of the background, the corrected pole figure is constructed in spherical coordinates by linear interpolation of the data collected on the equal area spherical net. The pole figure, plotted in Figure 2, clearly shows the expected radial symmetry, and confirms that the (0002) planes are preferentially oriented parallel to the sample surface.

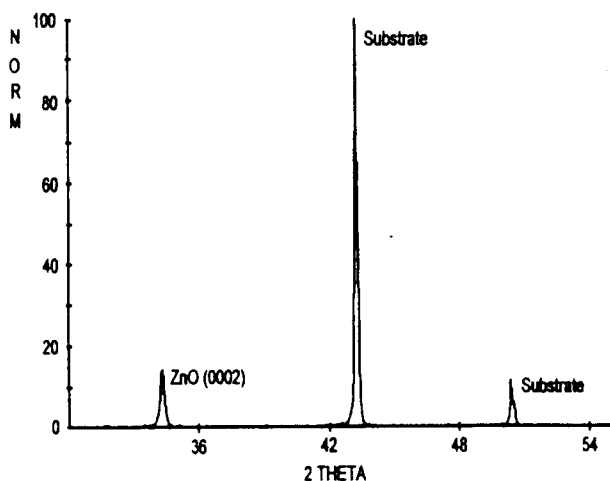


Fig. 1 - Diffraction Pattern for ZnO CVD Coating

After confirming the suspected (0002) texture, the partial pole figure can be calculated using the average of all intensity values collected at each increment of tilt, ϕ . A plot of the intensity vs. tilt is shown in Figure 3. The intensity decreases rapidly from a maximum of nominally 9000 counts per second (cps) at $\phi=0$, to negligible diffracted intensity beyond approximately 45 deg. Finally, the average intensity collected is totaled and the fraction of the total intensity between 0 deg. and each angle of tilt is calculated and plotted.

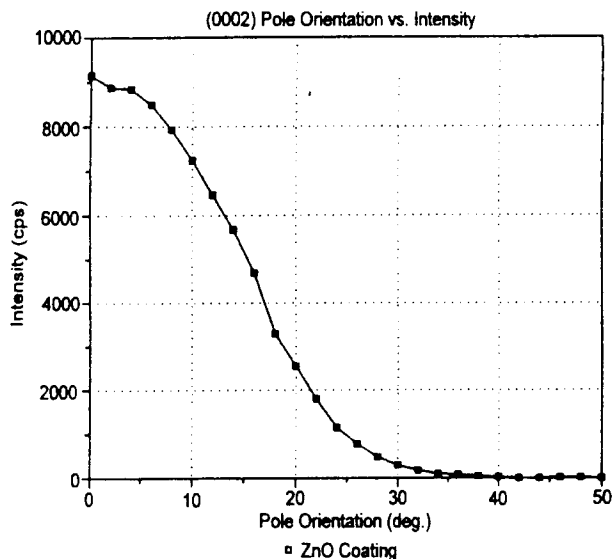


Fig. 3 - Radial (0002) Pole Density Distribution for CVD ZnO Film

The partial pole figure, plotted in Figure 4, indicates a nearly linear relationship between pole fraction and tilt between 0 and 10 deg. Approximately 56% of the crystals are oriented between 0 and 10 deg. from the surface, 91% between 0 and 20 deg., and nearly 100% between 0 and 30 deg.

The slope of the linear portion of the partial pole figure as it approaches 0 deg. is indicative of the severity of the texture present, and can be used to compare the degree of texture in different specimens. The slope increases rapidly as the spread in the distribution of crystal planes parallel to the sample surface diminishes. For the case of the ZnO coating, the slope is nearly constant between 0 and 10 deg. and is equal to 5.6%/deg.

Variations in the slope of the partial pole figure can also reveal the presence of secondary textures. For example, if the (0002) crystals had some degree of preferred orientation at 20 deg., in addition to the main orientation at 0 deg., there would be a corresponding increase in the slope of the partial pole figure at 20 deg.

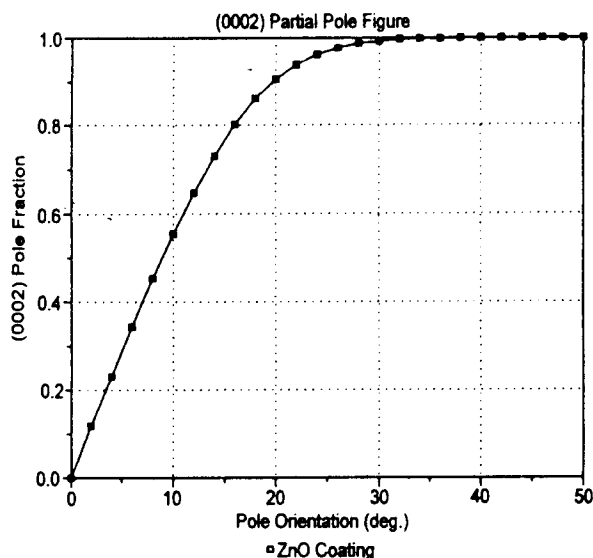


Fig. 4 - Partial (0002) Pole Figure for CVD ZnO Film

If you are interested in partial pole figure analysis or texture analysis, or would like to discuss a specific application of this technique, please contact Tom Easley, Research Engineer, at (513) 561-0883.

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References

1. Yang, L., and R.G. Hudson. "CVD of Refractory Metals." American Nuclear Society (1967): 41-43.

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New Promotions

Lambda Research is proud to announce the recent promotions of two employees. Thomas Easley, formerly Senior Laboratory Technician in the Quantitative/Texture Analysis Laboratory, has been promoted to Research Engineer. Douglas Hornbach, formerly Senior Laboratory Technician in the Stress Laboratory, has also been promoted to Research Engineer. These recent promotions were announced at the quarterly associates meeting.

Direct and Inverse Pole Figures in Zirconium and Hafnium Alloys

Lambda Research has been approved by Bettis Nuclear Laboratories to provide inverse pole figures on zirconium and hafnium alloys. Direct pole figures and ODF analysis have also been performed for several other nuclear applications. Contact: Tom Easley