ADVANTAGES OF USING 2D DETECTORS FOR RESIDUAL STRESS MEASUREMENTS

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The diffracted beams from a polycrystalline sample form a series of cones corresponding to each set of lattice planes. Introducing a stress into the sample distorts the diffraction cone shape. An area detector can be considered as a plane intersecting the diffraction cones. The authors of this paper have previously developed a new theory in which a direct relation between the stress tensor and the diffraction cone distortion is given. In this paper, the advantages of using 2D detectors for residual stress measurement based on both the conventional "sin²ψ" theory and the new 2D theory are summarized. Some application examples are also discussed based on the experimental results with a Bruker Hi-Star area detector. The experiments have shown that there are many advantages to use 2D detector for stress measurement, including high measurement speed, high accuracy, and virtual oscillation for large crystals and textured samples.

1 Introduction

In recent years, two-dimensional (2D) detectors are extensively used for single crystal diffraction and small angle scattering [1]. There is also an increasing trend in using two-dimensional detectors for x-ray powder diffraction. It has been reported that two-dimensional detectors have advantages in many x-ray powder diffraction applications, such as quantitative texture analysis, micro-area phase identification, and polymer crystallinity [2-4]. At present, x-ray diffraction residual stress determination based mainly on the one-dimensional (1D) diffraction profile measured with a scanning point detector or a one-dimensional position-sensitive detector (PSD). The availability of 2D technology has rarely benefited the residual stress field. The main reasons are limited experimental experience and lack of theory to take the full advantage of the 2D diffraction data. In this paper, we first compare the differences between 1D technique and 2D technique in geometric configuration, stress measurement theory and diffraction data structure. Then the advantages of using 2D detectors for residual stress measurement based on both the conventional "sin²ψ" theory and the new 2D theory are discussed.

2 Theory

2.1 Conventional Theory

In the conventional stress measurement, one diffraction profile is measured at each sample orientation defined by two angles, φ and ψ. The 2θ-shift determined by that diffraction profile serves as a strain gage in the specific direction. The relationship between each strain measurement and the strain tensor in the sample is defined by a so-called fundamental equation of x-ray strain determination [5].

\[ \varepsilon_{\phi\psi} = \varepsilon_{11} \cos^2 \phi \sin^2 \psi + \varepsilon_{12} \sin 2\phi \sin^2 \psi + \varepsilon_{22} \sin^2 \phi \sin^2 \psi + \varepsilon_{33} \cos^2 \psi \]

where \( \varepsilon_{\phi\psi} \) is the measured strain in the orientation defined by φ and ψ angles and \( \varepsilon_{11}, \varepsilon_{12}, \varepsilon_{22}, \varepsilon_{13}, \varepsilon_{23} \) and \( \varepsilon_{33} \) are strain tensor components in the sample coordinates S, S, S as shown in Figure 1a. In the equation, one 2θ-shift value (d-spacing change) is considered at each sample orientation (ψ, φ), this is...
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suitable to the stress measurement with point detectors or one-dimensional position-sensitive detectors. In
the conventional methods, φ rotation axis is the sample surface normal S3 and ψ-tilt is achieved by either ω
rotation on an Ω-diffractometer (iso-inclination) or ψ rotation on a Ψ'-diffractometer (side-inclination) as
is shown in Figure 1b.

![Figure 1](image)

Figure 1. Configurations for conventional stress measurement method. (a) The relation between the
measured strain ε and the sample coordinates S1S2S3. (b) Two kinds of ψ-tilt.

2.2 New 2D Theory

The diffracted beams from a polycrystalline sample form a series of cones corresponding to each set of
lattice planes, as is shown in Figure 2a. The incident x-ray beam lies along the rotation axis of the cones.
The apex angles of the cones are determined by the 2θ given by the Bragg equation. The χ angle is the
azimuthal angle from the origin at the 6 o'clock direction with rotation axis along the incident x-ray beam.
The χ angle defines each diffracted beam on the diffraction cone. The diffraction cones from an unstressed
polycrystalline sample are regular cones in which 2θ is independent of χ and 2θ = 2θ0. Introducing a stress
into the sample distorts the diffraction cone shape so that 2θ becomes a function of χ, 2θ = 2θ(χ). This
function is uniquely determined by the stress tensor and the sample orientation.

Figure 2b shows the sample orientation angles ω, ψ, and φ. S1S2S3 are sample coordinates with S1S2 on the
sample surface plane and S3 as surface normal. At ω = ψ = φ = 0, S1 is in the opposite direction of the
incident x-ray beam, and S2 points up and overlaps with ω-axis. The ω-axis is fixed on the laboratory
coordinates, ψ is a rotation above a horizontal axis and φ is a left-hand sample rotation about its normal
S3. ψ-axis varies with ω rotation and φ axis varies with ω and ψ rotation. Since the χ symbol has been
used for the azimuthal angle on the diffraction cones in this paper, we use χe to represent the χ rotation in
the 4-circle goniometer. ψ and χe have the same axis but different starting position and rotation direction,
and χe = 90°, ψ.

The surface of the 2D detector can be considered as a plane intersecting the diffraction cones. Figure 3
shows the diffraction data collected on 2D detectors, α is the detector swing angle. When imaged on-axis
(α = 0°) the conic sections appear as circles. When the detector is at off-axis position (α ≠ 0°), the conic
section may be an ellipse, parabola, or hyperbola. For convenience, all kinds of conic sections will be
referred to as diffraction rings hereafter in this paper. All diffraction rings collected in a single exposure will be referred to as one frame. The 2D detector image (frame) is stored as intensity values on a grid of pixels, (1024x1024 pixel on Bruker GADDS area detector). The corresponding 2θ and χ values on each pixel are also given by the 2D diffraction system. The diffraction profile on a particular χ line can be calculated from the 2D image by χ-integration within a given χ range. The peak position at each χ angle can be determined from the diffraction profile by peak fitting method. The number of data points from one ring depends on the total χ range and χ integration steps. The diffraction cone distortion due to stresses is recorded as a function 2θ(χ).

Figure 2. (a) The diffraction cones from an unstressed polycrystalline sample and the diffraction cone distortion due to stresses. (b) Sample orientation in terms of ω, ψ and φ angles.

Figure 3. The diffraction rings collected on area detectors on-axis or off-axis.

The strain tensor in the sample coordinate system $S_1S_2S_3$, the sample orientation (ω, ψ, φ), and the diffraction data (χ, 2θ) are related by the following expression.

$$f_{ij}ε_{ij} + f_{12}ε_{12} + f_{22}ε_{22} + f_{13}ε_{13} + f_{32}ε_{32} + f_{33}ε_{33} = \ln\left(\frac{\sin θ_{0}}{\sin θ}\right)$$

(2)

where $f_{ij}$'s are strain coefficients determined by the equations listed in Table 1.
Table 1. Equations for Calculation of Strain Coefficients $f_i$  

<table>
<thead>
<tr>
<th>Strain Coefficient</th>
<th>$f_{11}$</th>
<th>$f_{12}$</th>
<th>$f_{22}$</th>
<th>$f_{13}$</th>
<th>$f_{23}$</th>
<th>$f_{33}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equations</td>
<td>$A^2$</td>
<td>$2AB$</td>
<td>$B^2$</td>
<td>$2AC$</td>
<td>$2BC$</td>
<td>$C^2$</td>
</tr>
<tr>
<td>$a = \sin b \cos \omega + \sin \chi \cos b \sin \omega$</td>
<td>$A = a \cos \phi - b \cos \psi \sin \phi + c \sin \psi \sin \phi$</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>$b = -\cos \chi \cos b$</td>
<td>$B = a \sin \phi + b \cos \psi \cos \phi - c \sin \psi \cos \phi$</td>
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<td></td>
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<tr>
<td>$c = \sin b \sin \omega - \sin \chi \cos \phi \cos \omega$</td>
<td>$C = b \sin \psi + c \cos \psi$</td>
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</tr>
</tbody>
</table>

Equation (2) is the fundamental equation for strain measurement by diffraction with 2D detectors, which gives a direct relation between the diffraction cone distortion and the strain tensor. Since it is a linear equation with six unknowns, in principle, the strain tensor can be solved with six independent $(\chi, \theta)$ data points. The least squares method can be used to solve the strain or stress tensor with very high accuracy. For isotropic materials, there are only two independent elastic constants, Young’s modulus $E$ and Poisson’s ratio $\nu$ or x-ray elastic constants $\frac{1}{2}S_2 = \frac{1}{E}(1 + \nu)$ and $S_1 = -\nu \div E$. Then the stress tensor can be calculated from

$$p_1 \sigma_{11} + p_2 \sigma_{12} + p_3 \sigma_{13} + p_{22} \sigma_{22} + p_{23} \sigma_{23} + p_{33} \sigma_{33} = \ln \left( \frac{\sin \theta_n}{\sin \theta} \right)$$

where $p_i = \begin{cases} (\sqrt{E})[(1 + \nu) f_{ii} - \nu] = \frac{1}{2} S_2 f_{ii} + S_i & \text{if } i = j \\ (\sqrt{E}(1 + \nu) f_{ii} = \frac{1}{2} S_2 f_{ii} & \text{if } i \neq j \end{cases}$

Figure 4. Relationship between diffraction ring on 2D detectors and 1D detector on diffractometer plane.

2.3 Relationship between Conventional Theory and 2D Theory

In order to find the relationship between the conventional theory and the new 2D theory, we first compare the configurations used for data collection in both cases. The conventional diffraction profile is collected with a point detector scanning in the diffractometer plane or a position sensitive detector mounted in the diffractometer plane. The 2D diffraction data consists of diffracted x-ray intensity distribution on the detector plane. The intensity distribution along any line defined by a fixed $\chi$ is a diffraction profile analogous to the data collected with conventional diffractometer. Figure 4 shows the relation between 2D
detector and conventional detector. The diffraction profiles at $\chi = 90^\circ$ and $\chi = -90^\circ (=270^\circ)$ on the 2D detector are equivalent to the diffraction profiles collected in the conventional diffractometer plane. Therefore, one can use diffraction profiles at $\chi = 90^\circ$ and $\chi = -90^\circ$ on a 2D detector to imitate a conventional diffractometer.

In theory, it has been proved that the conventional fundamental equation is a special case of the 2D fundamental equation [8]. In the same way, a conventional detector can be considered as a limited part of a 2D detector. Depending on the specific condition, one can choose either theory for stress measurement when a 2D detector is used. If the conventional theory is used, one has to get a diffraction profile at $\chi = 90^\circ$ or $\chi = -90^\circ$, this is normally done by integration the data in a limited $\chi$ range. The disadvantage is that only part of the diffraction ring is used for stress calculation. When the new 2D theory is used, all part of the diffraction ring can be used for stress calculation.

3 Advantages of Using 2D Detectors

There are many advantages of using 2D detectors for residual stress measurement, no matter if the conventional "$\sin^2 \psi$" theory or the new 2D theory is used. Some application examples are discussed here from the experimental results with a Bruker Hi-Star area detector. The speed of data collection with an area detector can be $10^4$ times faster than with a point detector and about 100 times faster than with a linear position-sensitive detector. And most important is the large dynamic range and simultaneous collection of 2D information. The experiments have shown that advantages to use 2D detectors for stress measurement include, but not limited to, high sensitivity, high measurement speed, high accuracy; virtual oscillation for large crystals and textured samples.

3.1 High Sensitivity – Residual Stress from The Inside Surface of A Small Spring

This is an example of residual stress measurement with a GADDS Microdiffraction System. The residual stress on the inside surface of a small spring (coil diameter -10mm, wire diameter 1mm and coil pitch 4mm) was measured with Cr-K$\alpha$ radiation and 0.3mm collimator. The spring was made of precipitation stainless steel 17-7PH. The (211) diffraction ring of the $\alpha$-phase was used for stress measurement. Fig 5 shows the laser spot on the inside surface of the spring wire. When the laser spot overlaps with the center of the crosshair the sample surface is aligned to the goniometer center. The x-ray beams can pass through the gap between spring wires so the residual stress can be measured nondestructively.

Figure 5. The images from the laser video sample alignment system. (a). Sample alignment with laser spot and crosshair. (b). X-ray beam hit the inside surface (it appears like outside surface due to the depth of focus) of the spring wire by passing through the gap between the wires.
Fig 6. shows one of the measured frames with chi-integrated profile. The χ-integration gives a sound profile although the diffracted intensity is quite low on the 2D frame. The results are listed in Table 2. The ψ tilt is achieved by Ω scan. The residual stress values determined in scans of 7 and 19 steps agree very well. The 19 point measurement has a lower standard deviation, about 3.5%.

![Image of a measured frame with chi-integrated profile. The green broken line box defines the chi integration region. The blue broken lines indicate the shadow of the wires.](image)

Figure 6. A measured frame with chi-integrated profile. The green broken line box defines the chi integration region. The blue broken lines indicate the shadow of the wires.

| Table 2. Residual stress measurement results of the inside surface of a stainless steel spring. |
|-----------------------------------------------|---------------|
| Number of frames | 7 | 19 |
| ψ angles and steps | -45° to 45°, 15° steps | -45° to 45°, 5° steps |
| Data collection time | 14 minutes | 38 minutes |
| Measured stress | -864 (± 48) MPa | -875 (± 31) MPa |

3.2 High Accuracy – 2D Method vs. Conventional Method

The residual stresses on a hardened carbon steel roller were measured by the conventional method and the new 2D method [7]. The roller is a cylinder 1” long and 6/8” in diameter. The stress data was taken from the center of the roller end. A total of 7 frames were taken with ω angles at 33, 48, 63, 78, 93, 108, 123°
with Cr-Kα radiation. The (211) ring in the χ range of 67.5° to 112.5° was used for stress analysis. First, the frame data was integrated along the χ angle in the interval of Δχ=5°. A total of 9 diffraction profiles were obtained from the χ integration. The peak position 2θ at each χ angle was then obtained by fitting the profile with Pearson VII function. The data points at χ=90° from 7 frames, a typical data set for Ω-diffractometer, are used to calculate stress with the conventional sin²ψ method. In order to compare the statistic error between different number of data points, different number of data points (3, 5, 7 and 9) are used to calculate stress with the new 2D equation. The results from the conventional method and the new 2D method are compared in Figure 7. The measured residual stress is compressive and the stress values from different methods agree very well. With the data taken from the same measurement (7 frames), the new 2D method gives lower statistical error and the error decreases with increasing number of data points from the diffraction ring.

![Graph showing comparison of stress methods]

Figure 7. Comparison of the conventional method and the new 2D method with different number of data points. (a) Data points taken from the diffraction ring; (b) Measured stress and standard deviation for different methods and different number of data points.

### 3.3 High Speed – Biaxial Stress Tensor from Seven Minute Data Collection

This is an example of stress tensor measurement from data collected in 7 minutes. A Bruker GADDS Microdiffraction System with the following configuration: Cr-Kα radiation (λ=2.2897Å); generator power 35kV/50mA; 0.5mm collimator with front pinhole only. The stress measurement parameters are: 2θ₀ =156°(211), E=210000 MPa, ν = 0.28, Arx = 1.49. The sample is an almen strip shot peened for 30 minutes with S170 cast steel shot. Seven one-minute frames were taken at the following ω and φ angles.

<table>
<thead>
<tr>
<th>Frame #</th>
<th>.000</th>
<th>.001</th>
<th>.002</th>
<th>.003</th>
<th>.004</th>
<th>.005</th>
<th>.006</th>
</tr>
</thead>
<tbody>
<tr>
<td>ω angle</td>
<td>57°</td>
<td>72°</td>
<td>87°</td>
<td>102°</td>
<td>117°</td>
<td>132°</td>
<td>147°</td>
</tr>
<tr>
<td>φ angle</td>
<td>0°</td>
<td>45°</td>
<td>90°</td>
<td>0°</td>
<td>45°</td>
<td>90°</td>
<td>0°</td>
</tr>
</tbody>
</table>

Each diffraction ring are fitted to 11 data points from χ =62.5° to 117.5° with Δχ=5°. The calculated biaxial stress tensor components are: σ₁₁ = -603±20 MPa, σ₁₂ = -568±50 MPa and σ₁₂ = 12±40 MPa. The data points and diffraction ring corresponding to the calculated stresses are displayed for one of the frames (#006) in Figure 8.
3.4 Virtual Oscillation – Residual Stress from Samples with Large Grain Size

In the case of materials with large grain size or microdiffraction with a small x-ray beam size, the diffraction profiles are distorted due to poor counting statistics. To solve this problem with conventional detectors, some kind of sample oscillations, either translation oscillations or angular oscillations are used to bring more crystallites into diffraction condition. In another words, the purpose-of oscillations is to bring more crystallites in the condition that the normal of the diffracting crystal plane coincides with the instrument diffraction vector. For 2D detectors, when the $\chi$-integration is used to generate the diffraction profile, we actually integrate the data collected in a range of various diffraction vectors. The angle between two extreme diffraction vector is equivalent to the oscillation angle in a so-called $\psi$-oscillation. Therefore, we may call this effect as “virtual oscillation”. Figure 9 shows the relation between the $\chi$-integration range, $\Delta \chi$ and the virtual oscillation angle, $\Delta \psi$. The $2\theta$ value of the $\chi$-integrated profile is an average over the Debye ring defined by the $\chi$-range. The average effect is over a region of orientation distribution, rather than a volume distribution.

Figure 9. The relation between the $\chi$-integration range, $\Delta \chi$ and the virtue oscillation angle $\Delta \psi$. 
The virtual oscillation angle $\Delta \psi$ can be calculated from the integration range $\Delta \chi$ and Bragg angle $\theta$.

$$\Delta \psi = 2 \arcsin[\cos 6 \sin(\Delta \chi / 2)]$$  \hspace{1cm} (4)

For example, Figure 10 is a frame taken from a stainless steel with large grain size. If we integrate from $\chi=80^\circ$ to $100^\circ$, $\Delta \chi = 20^\circ$, $\theta = 64^\circ$, the virtual oscillation angle $\Delta \psi = 8.7^\circ$. In the conventional oscillation, mechanical movement may result in some sample position error. Since there is no actual physical movement of the sample stage during data collection, the virtual oscillation has no such a problem.

Figure 10. A diffraction frame taken from a stainless steel. The virtue oscillation by $\chi$-integration over $\Delta \chi = 20^\circ$ gives a smooth diffraction profile.

Figure 11. Frames collected from samples with various degrees of texture, from random powder to very strong texture. With the very strong textured sample, conventional diffractometer may miss the diffraction ring.
3.5 Texture – Residual Stress from Textured Samples

The diffraction frames collected with a 2D detector contains both stress and texture information. Two functions can be derived from the diffraction ring. One is the peak position as a function of $\chi$, $2\theta = 2\theta(\chi)$, which is uniquely determined by the stress tensor and the sample orientation. Another is the integrated intensity as a function of $\chi$, $I = I(\chi)$, which is determined by the sample texture. Figure 11 shows four frames collected from samples with no texture, weak texture, strong texture and very strong texture. For the case with very strong texture in this example, the conventional diffractometer using a scanning point detector or PSD will miss the diffraction ring, so as not to be able to measure the diffraction peak. For mild texture, the virtue oscillation can be used for the stress calculation. For strong texture, the diffraction profiles integrated over a large $\Delta \chi$ may not accurately represent the angular position of measurement. In this case, the new 2D method should be used for stress calculation from the diffraction profiles generated at various $\chi$ angles with a relative small $\Delta \chi$. Since the diffraction data includes both stress and texture information, 2D detector also make it possible to measure stress and texture simultaneously. This is necessary for corrections on the elastic anisotropy caused by texture.

3 Conclusions

Since 2D detectors can observe the whole or a part of the diffraction ring, compared with a single diffraction profile from 1D detectors, there are many advantages of using 2D detectors for residual stress measurement. A new 2D theory gives a direct relationship between the stress tensor and the diffraction conic section distortion. The speed of data collection with a 2D detector is faster than with a point detector or a PSD. The experiments have shown that 2D detectors can measure stress with high sensitivity, high speed and high accuracy. The virtual oscillation can deal with samples with large crystals and/or texture. Simultaneous measurement of stress and texture is also possible since 2D data consists of both stress and texture information. Stress measurement using 2D detector is based on the new 2D theory and 2D detector technology. With the availability of 2D detectors with large area, fast and higher resolution, and with more experimental experience, we expect to find more advantages of using 2D detectors for stress measurement.

References