A Calibration Procedure for Position Sensitive Detectors Used in Neutron Residual Strain Mapping

Xiaojing Zhu and Camden R. Hubbard

High Temperature Materials Laboratory
Oak Ridge National Laboratory
P.O. Box 2008, Oak Ridge, TN 37831-6064, USA
(phone: 423-574-4472, e-mail: hubbardcr@ornl.gov)

ABSTRACT

A position sensitive detector (PSD) coupled with a multi-channel analyzer (MCA) is a desirable neutron detection system in residual strain mapping, which requires both high sensitivity and high speed in data acquisition. While this PSD/MCA system can be used for strain measurement, it needs to be calibrated and the calibration parameters stored for later retrieval in data analysis. The relationship between precision of peak location and counting statistics is an important factor in optimizing measurement time. Generally, for diffraction near 90° 2θ a precision of 0.01° 2θ or smaller is required. Such precision can be achieved with the PSD/MCA system by acquiring a peak intensity of at least 100 counts above background.

INTRODUCTION

Residual stresses, which affects mechanical behavior and life expectancy, exist in most engineering structures. A good understanding of the stress distribution in materials can lead to improved design and life expectancy of engineering structures. Diffraction methods have long been used for strain measurement and stresses are obtained from the measured strains with Hooke’s law and knowledge of the diffraction elastic constants. The neutron diffraction method, with its much greater penetration depth than x-rays, can be used in nondestructive residual strain characterization inside materials, such as steel and aluminum.1,2,3 The advantage of nondestructively obtaining the triaxial strain values is that the strains, their orientation, and their gradients can be measured more realistically and accurately by neutron diffraction methods compared to depth profiling via hole drilling. With the increase in computing power of modern computer system, numerical models have been developed to simulate the stress state/distribution in engineering structures. Hence, the stresses determined experimentally can be used to improve, verify and/or validate these numerical models.

The use of a position sensitive detector allows rapid acquisition of the complete neutron diffraction peak profile without motion of the detector arm thereby avoiding possible instrument errors due to movement of 2θ. Strain mapping requires precision in lattice spacing on the order of one part in ten thousand, which for neutron diffraction peaks near 90° 2θ, corresponds to a precision in peak position of better than 0.01° 2θ. One goal of this study is to determine the peak counts in the profile minus background counts to achieve such precision.

The PSD/MCA system needs to be calibrated before any meaningful data can be taken. A calibration process generally involves (a) testing for sensitivity across all channels of a PSD, (b) establishing the relationship between the detector channels and the angular positions, and (c) evaluating the zero offset of the PSD and the neutron wavelength. The calibration parameters are detector channel sensitivities (s), detector angular change (deg) per channel (k), detector zero offset (2θ₀) and neutron wavelength (λ). Furthermore, the stability and sensitivity of the PSD as well as the relationship between the peak intensity and the estimated standard deviation (ESD) in...
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peak position need to be characterized. The ORNL Neutron Residual Stress Facility (NRSF) procedure for PSD/MCA calibration and typical calibration results are discussed in this paper. This procedure has been implemented in Labview™ for convenient data collection and processing.

THE PSD/MCA DETECTOR SYSTEM

The detector used is an ORDELA 1155N helium-3 neutron detector (Fig. 1). For each detected neutron, the $^3$He(n,p) reaction produces about $2.6 \times 10^4$ free electrons and gas ions in the active volume near the interaction position. The specifications for the detector system are listed as follows:

- Counting gas: $^3$He-CF$_4$ at 700 kPa
- Bias voltage: ~3.1 kV
- Construction: 2.5 mm thick Al window for low scattering and faster data collection
- Active volume: 100 x 40 x 20 mm$^3$
- Spatial resolution: ~1 mm
- Pixels: 512 channel MCA sampling
- Efficiency: 43% at 1Å, 67% at 2Å and 81% at 3Å
- Count rate capability:
  - At 10% loss: $2 \times 10^4$ neutrons/sec
  - Maximum: $10^6$ neutrons/sec

Figure 1. Position sensitive neutron detector (ORDELA 1155N) is used at NRSF.

PSD CALIBRATIONS

Three major steps are taken to obtain the calibration parameters specified in the introduction section: (I) determine the detector intensity sensitivity, (II) determine the channel to angular relationship, and (III) determine the detector zero shift ($\theta_0$) and neutron wavelength. Although the wavelength is not used directly in the data analysis of strain, it is obtained and used to calculate the fitting residual in the parameter fitting process and enables conversion of $\theta_{corr}$ to d-spacing.
Step I: Determination of detector sensitivity

The intensity sensitivity across the PSD can be affected by the detector construction and electronic setting of the multi-channel analyzer. An uneven sensitivity across the detector adds artifacts to the diffraction peak profiles collected. To eliminate these artifacts the sensitivity of each PSD/MCA channel needs to be determined and used to normalize the channel sensitivities.

Detector sensitivity is determined by using scattered neutrons from a paraffin block. The neutron beam from the reactor core is made monochromatic by the (11.0) plane of a beryllium single crystal. The incident beam is collimated by a 2x10 mm incident slit and is uniformly scattered by the paraffin, sized at 40x60x8 mm^3. The spectrum is collected by the PSD/MCA system controlled by a data collection program. A typical spectrum of the neutron flooded detector is shown in Fig. 2. To obtain good counting statistics, a large number of counts (up to ~100k) is necessary. Generally it is observed that some channels at the two ends of a neutron PSD are inactive (about 60 channels on each end). Only the sensitivity of each active channel needs to be determined. The sensitivity spectrum in Fig. 2 is quite uniform except for some oscillations between channel numbers 220 and 300. This “ringing” is reproducible and is attributed to the detector electronics.

To set a reference intensity for normalization, the medium intensity of the detector in the range of the active channels (I_m) is obtained. Then the detector channel sensitivities (s_i) are calculated as

\[ s_i = I_i / I_m \]  

where I_i is the number of counts at channel i. Thus, diffraction peak profiles subsequently measured with the detector can be normalized for detector sensitivity by

\[ I_{i(norm)} = I_{i(meas)} / s_i \]  

Figure 2. A typical spectrum of a detector flood.

Step II: Determination of the channel to angular relationship

The PSD/MCA system records diffraction profiles as counts versus channel number and so the relationship between a channel number and the angular position needs to be established. With high quality systems this relationship is very close to a straight line. Assume the PSD is mounted on the 2θ-arm so that higher channel numbers of the PSD/MCA correspond to higher
2θ angles. For purposes of this paper, assume the PSD response is linear across the detector. The diffraction profile positions in angular form are (Fig. 3):

$$2\theta_{\text{meas}} = a_0 + k i_{\text{meas}}$$  \hspace{1cm} (3)

where $2\theta_{\text{meas}}$ is the angular profile position and $i_{\text{meas}}$ is the measured profile position in PSD channels, $a_0$ is the angle corresponding to channel $i = 0$ of the PSD and $k$ is the linear slope in degrees 2θ per PSD/MCA channel. If we define the angle of the PSD’s center channel, $i_c$, as the PSD position, $2\theta_{\text{det}}$ (a different definition only results in a difference in detector zero offset), we obtain

$$a_0 = 2\theta_{\text{det}} - k i_c$$  \hspace{1cm} (4)

In theory, the parameter $k$ can be obtained by measuring the shift in peak position on the PSD/MCA corresponding to a small angular shift of the detector. In practice, to improve the determination of $k$, a set of peak profiles are acquired at consecutively shifted detector positions. Figure 4 shows the superposition of many recorded spectra of a nickel (311) peak. The size of each shift used here is 0.2°. For a PSD covering a width of ~5°, about 20 shifted profiles can be collected. The specimen is nickel powder sealed in a 2 mm diameter, 40 mm long quartz tube. Either Ni (111) or (311) peaks can be used because of their high intensities. The centroids of the peak profiles are determined by fitting Gaussian functions to the profiles after normalizing the detector channel sensitivity (Eq. 2). The detector angular positions, $2\theta_{\text{det}}$, are then plotted against the corresponding peak positions (in channel numbers) and a linear least squares fit is used to obtain the slope, $k'$ (angular change per channel). The quality of fit also reveals the channel linearity of the PSD/MCA and the accuracy of detector positioning. For the detector used at NRSF, a linear fit typically yields a goodness of fit of 0.9997 or better.

Because the peak registered in the PSD/MCA moves to a lower channel position when the PSD is shifted to a higher angle, the $k'$ obtained by detector shifting and $k$ of Eq. 4 have the same magnitude but opposite signs, i.e.,

$$k = -k'.$$  \hspace{1cm} (5)

Substituting Eq. 4 and 5 into Eq. 3, we have

$$2\theta_{\text{meas}} = 2\theta_{\text{det}} + k' (i_c - i_{\text{meas}})$$  \hspace{1cm} (6)

Figure 3. Illustration of channel to angular conversion where $i_c$ is the center channel number of the PSD, $i_{\text{meas}}$ is the measured peak position in channels, and $2\theta_{\text{det}}$ is the angle of the detector arm.
Step III: Determine the detector zero offset (2\(\theta_0\)) and wavelength (\(\lambda\))

The relationship between \(2\theta_0\) and \(\lambda\) is given by the Bragg equation

\[
d_{hkl} = \frac{\lambda}{2 \sin(\theta - \theta_0)},
\]

where \(d_{hkl}\) is the lattice spacing of a diffraction line and \(\theta = \theta_{\text{meas}}\) in Eq. 6. Since the lattice parameters of the standard nickel powder specimen are known, the \(\lambda\) and \(\theta_0\) can be obtained by minimizing

\[
\chi^2 = \sum (d_{hkl} - d'_{hkl})^2
\]

using nonlinear parameter fitting. Here \(d'_{hkl}\) is the lattice spacing calculated from the known lattice parameter. In the case of a cubic lattice

\[
d'_{hkl} = \frac{a}{(h^2 + k^2 + l^2)^{1/2}},
\]

where \(a\) is the lattice parameter. The necessary data for this calibration step are profiles at the first five (fcc) nickel lines, i.e., (111), (200), (220), (311) and (222) acquired as a function of detector position \(2\theta_0\). Applying corrections steps I & II and then profile fitting yields the \(2\theta_{\text{meas}}\) for each measured peak. A Levenberg-Marquardt algorithm for non-linear least square fitting is then used to obtain \(\theta_0\) and \(\lambda\) via Eq. 8.

Implementation of the procedure

The above calibration procedure, along with the data acquisition and processing for neutron strain mapping, has been implemented in Labview\textsuperscript{TM}3 on a Macintosh computer. The measured raw data are stored in a *rd data log file (the '*' here is a wild card character which can be substituted by any characters, or string of characters).

For Step I, the s's are calculated and saved in a file whose name ends with .cint for further use in channel sensitivity normalization for collected raw data profiles. In Step II, the intensity in each PSD/MCA channel is normalized using the data in *.cint file and then the profile is stored in a *.int file. This intensity corrected profile is subsequently fitted with a Gaussian function. The resulting profile parameters, such as peak position and full width at half maximum (all in channels), peak height (less background) and background shape parameters (linear or quadratic), are saved in a binary *.pk file. These parameters are also saved in an ASCII file (*.apk). The channel number to angular conversion is obtained from the slope of a linear least squares fit for detector angle versus peak channel position.
Step III of the calibration process is implemented as follows: The raw profiles of the first five nickel lines are normalized for channel intensity sensitivity and converted from channel distribution to angular distribution. The resulting profiles are stored in a *.ang file. The peak positions, FWHM and other parameters, as well as their standard deviations are obtained from Gaussian profile fitting, and are saved in a *.pk file. These data are then substituted into Eq. 8 and a Levenberg-Marquardt algorithm is used to obtain $\theta_0$ and $\lambda$ by minimizing the $\chi^2$ value. The output is stored in a *.czer file.

To summarize, the calibration parameters generated in Steps I-III are stored in files *.cint, *.cang and *.czer. After this it is only necessary to apply these calibration data to the acquired raw data files as follows:

a) Apply intensity compensations;
b) Convert the channel numbers to angles;
c) Apply $\theta_0$ correction using

$$2\theta_{\text{cor}} = 2\theta_{\text{meas}} - 2\theta_0.$$  \hspace{1cm} (9)

### CALIBRATION RESULTS

In addition to the calibration parameters obtained in Step III, the fitting residual ($\Delta 2\theta$) is also obtained. An example output is shown in Fig. 5. Here $\Delta 2\theta$ is defined as

$$\Delta 2\theta = 2\theta_{\text{cor}} - 2\theta_{\text{calc}}$$  \hspace{1cm} (10)

$$2\theta_{\text{calc}} = 2 \arcsin(\lambda / 2d_{hkl})$$  \hspace{1cm} (11)

where $\lambda$ is the wavelength obtained in the parameter fitting and $d_{hkl}$ is the $d$-spacing calculated from the reference lattice parameter. For a recent calibration (Fig. 5), the fitting residual ($\Delta 2\theta$) is less than $\pm0.01^\circ$ for all of the five nickel lines used (over a $2\theta$ range of 42$^\circ$ to 97$^\circ$). This indicates that we can expect to achieve a similar $2\theta$ accuracy in the $d$-spacing measurements using these calibration parameters.

![Figure 5](image.png)

**Figure 5.** Residuals of the nonlinear parameter fitting. Plotted in $\Delta 2\theta = 2\theta_{\text{cor}} - 2\theta_{\text{calc}}$.

### ELECTRONIC STABILITY AND COUNTING STATISTICS

The electronic stability of the detector system is critical to the accuracy of the strain measurements, because strain is determined from the difference between the diffracted peak position in a stressed specimen and that of a stress free reference. Thus, the greater the system stability the higher the accuracy of the strain determination. A series of tests was performed to
ascertain the electronic stability of the detector and electronics. Furthermore, to carry out the strain measurements efficiently, it is necessary to estimate the appropriate counting time that yields acceptable statistical uncertainty in the acquired data.

**Experiment**

The experiment to check stability and to determine the necessary PSD counting statistics was carried out by: (a) acquiring a peak profile repeatedly and (b) increasing the amount of counting time (0.5, 1, 2, 3, 4 and 8 minutes). The counting times were selected to yield a range of peak intensities (counts). Seven repeated measurements were made at each time level. The nickel (311) line was used in this experiment (the same specimen used in the PSD calibration). All peak profiles were calibrated using the calibration parameters obtained in the previous section. Each peak profile was then fitted with a Gaussian function to obtain peak height, peak position and peak FWHM. The result of the seven measurements at each counting time was averaged to obtain the average peak height, peak position and FWHM. The estimated standard deviation (ESD) from the average for each variable was also calculated.

**Results**

The peak height increased linearly with counting time (Fig. 6a). This verified detector linearity and short term electronic stability of the PSD/MCA system. The ESDs in the peak positions were less than 0.010°, 0.007° and 0.004° when peak heights were at least 50, 100 and 380 counts above the background, respectively (Fig. 6b). The decrease approximately follows an $N^{-1/2}$ behavior indicating that any electronic instability corresponds to less than 0.004° 2θ. The ESD decreases slowly with longer counting time. To achieve a precision in peak position of <0.01°, at least 100 counts over the background is sufficient. This 0.01° 2θ uncertainty corresponds to a 0.01% strain accuracy using a diffraction peak at 90° 2θ.

The average peak position as a function of counting time (or peak height indirectly) is shown in Fig. 6c. The vertical error bars are the typical ESD of the position from a single profile fitting. For the radiation used, the peak position of nickel (311) is determined to be 91.909±0.004° with 8 minutes counting time (380 counts peak height) and the FWHM is about 0.78° (Fig. 6d).

**SUMMARY**

The calibration procedure for a PSD/MCA system for neutron strain mapping has been developed and involves the following steps. First, determine the intensity response at each PSD channel. Second, establish the relationship between a shift in the detector arm and the apparent peak shift in channels. Third, assess the detector offset ($2\theta_0$) and neutron wavelength ($\lambda$) using nonlinear least square fitting. The first five diffraction peak profiles, (111), (200), (220), (311) and (222), of a nickel (fcc) standard powder were acquired with a PSD. For the neutron wavelength of about 1.53Å, the 2θ angles of these peaks range from ~42° to ~97°. The outcome of the calibration process is that the differences between observed and calculated 2θ angles of the first five fcc nickel lines are typically less than ±0.01° 2θ.

It was found that the ESD in peak position is less than 0.007° (2θ) as long as the peak intensity is higher than 100 counts over the background and the peak width and background are approximately equal to that observed for the Ni line. The long term detector and electronics stability and reproducibility were both found to be excellent, limited by the counting statistics, leading to an ability to measure strain rapidly with an estimated standard deviation of <$1 \times 10^{-6}$. 
Figure 6. Results of the counting statistics test with the PSD/MCA system: (a) average peak heights above background versus the counting time; (b) rms standard deviation of the peak position versus peak height; (c) average peak position versus counting time; (d) average peak full width at half maximum versus counting time.

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