

A study of factors that may influence the quality of residual stress measurements from X-Ray diffraction

Summary

XRD residual stress measurements have been performed on three engineering materials to assess the effect of changes to the test set-up and analysis method on the result. In particular a critical assessment of the values used during the residual stress calculation has been conducted and illustrations of the effect on the measured value and uncertainty reported.

Introduction

Within the European X-Ray diffraction (XRD) community work has been progressing to formulate a series of European standards relevant to XRD. These cover issues such as terminology, equipment, reference materials, general principles and residual stress measurement. In the case of residual stress measurement there are at present no international standards in existence, although there are a number of separate national documents that have been written and are used. This is soon to change with the publication of a new CEN standard for residual stress measurement by XRD. The objectives of this new CEN document as described by Delhez *et al* ^[1] is to improve the comparability of results obtained from different laboratories and test houses, and this will be achieved by the verification of instrument or products such that the testing is performed in comparable conditions, by using the same procedures and by using a common terminology.

With the need for validation and better agreement between laboratories and test houses it is important to appreciate the effects that different experimental factors will have on the final result, as, depending on the requirement for the residual stress measurement, these can influence the validity of the measured value.

The theory behind the XRD and the measurement techniques available have been well documented since Bragg first published his law on diffraction, this study therefore is not intended to address theoretical problems which have already been well addressed ^[2-6], but rather to examine and evaluate the experimental practicalities and analysis methods of routine XRD stress measurements.

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Material

For the purposes of this work measurements were performed on three materials representing a range of diffracting conditions, i.e. a ‘good’ diffracting material (shot peened spring steel), a ‘standard’ diffracting material (heat-treated aluminium) and a ‘poor’ diffracting material (ground titanium alloy).

All the measurements in this work were conducted using a Siemens D500 diffractometer using Cr-K α radiation.

Effect of Sample Position and Test Set up

The samples were subjected to a series of measurements designed to evaluate the relative sensitivity of the measurement technique to changes in the sample positioning and test set-up. The experiments were designed to investigate the effect on the results by introducing systematic changes in the test set up on a range of samples with fairly simple residual stress states i.e. homogenous across the surface. The tests were conducted to assess the following:

- Initial tests to establish the repeatability of the XRD technique, in terms of instrument only and instrument-operator repeatability.
- Effect of sample surface position (height offsets).
- Effect of changes in the data collection step size.
- Effect of altering the number of psi tilts performed.
- Effect of counting time.

Repeatability

To be able to critically assess the effect of changes in the experimental set up, it is first necessary to establish a measure of the repeatability of the measurements under identical conditions. To establish this, two series of tests were carried out on each sample. The first was to establish the repeatability of the equipment and data analysis using the system’s software. In these tests, each sample was placed on the stage and secured. The surface was carefully positioned so that it was at the centre of rotation of the goniometer, and ten repeat residual stress measurements made sequentially under the same conditions. At no

point during these repeat measurements was the sample moved and all the experimental parameters remained unaltered for all ten measurements. A second series of measurements was then carried out to establish the repeatability in the testing procedure. Within these tests the sample was removed from the machine between each test. Care was taken to ensure that the sample was replaced accurately so that the subsequent measurement was made in the same position as the previous one. All of the other test parameters remained constant. Details and results are given below.

Surface Position (Height Offset)

A number of tests were carried out where the height of the stage was offset from the centre of rotation of the goniometer. The offset range was $\pm 1\text{mm}$. The sample was not removed from the X-Ray machine between tests, and all other test parameters remained constant.

Data Collection Step Size

A series of measurements were made using different detector step sizes. The 2θ step size ranged from 0.01° up to 0.1° in increments of 0.01° . As before, the sample was not removed between tests and all other test parameters were kept constant.

Number of Psi Tilts

Without removing the sample, a succession of residual stress measurements were made using a different number of psi tilts. These ranged from 5 up to 21, but were always split between an equal number of positive and negative values.

Counting Time

The time that the detector counted at each step was also varied; the range was dependant on the material being measured.

Results

All the diffraction data was analysed using the average sliding gravity peak fitting method, and the results from the tests are summarised in Table I and Table II. For a more detailed report of these

measurements the reader is directed to Reference 7.

Table I Instrument and Instrument/Operator Repeatability for the three materials examined.

Sample	Results (MPa)	Instrument Repeatability	Instrument / Operator Repeatability
Shot Peened Steel	Mean	-547	-552
	Range	13	26
	Std dev	4	8
	CoV	0.7	1.4
Heat Treated Aluminium	Mean	-198	-202
	Range	6	8
	Std dev	2	3
	CoV	1.0	1.5
Ground Titanium	Mean	-418	-410
	Range	37	62
	Std dev	11	18
	CoV	2.6	4.4

The results in Table I showed that in all cases there was good repeatability in the measurement. The variation in the stress value as expressed by the coefficient of variation (CoV), was greater once there was the added influence of the operator, but remained within an acceptable level. For the two materials that exhibited good diffracting statistics, i.e. the steel and aluminium, the largest variation was found in the series of tests where the height offset was varied, where a CoV of 1.8% and 1.9% was measured respectively (Table II).

Table II Effect of data collection parameters on the variability in the measured residual stress values.

Sample	Results (MPa)	Height Offset	Step Size	Psi Tilts	Count Time
Shot Peened Steel	Mean	-566	-559	-559	-556
	Range	30	17	10	17
	Std dev	10	6	4	5
	CoV	1.8	1.1	0.7	0.9
Heat Treated Aluminium	Mean	-212	-205	-207	-206
	Range	14	8	6	8
	Std dev	4	3	2	3
	CoV	1.9	1.5	1.0	1.5
Ground Titanium	Mean	-420	-418	-418	-424
	Range	37	34	14	91
	Std dev	10	11	5	25
	CoV	2.4	2.6	1.2	5.9

In terms of the more difficult material with lower diffraction statistics, i.e. the ground titanium, the greatest scatter and uncertainty was obtained from varying the counting time. It should be noted that

generally for all the tests carried out on the ground titanium sample, it was not possible to get a well-defined peak for accurate analysis. This was largely due to insufficient counting statistics. With a longer count time, it is possible that the variation in the results reported for this material could be reduced and be brought more in line with the previous two materials.

Effect of Diffraction Data Analysis

Peak Fitting

The accuracy with which the diffraction peak can be located is vital for accurate residual stress analysis. The data sets previously discussed were analysed using several different methods of peak fitting, such that the variation in the values obtained could be assessed. The peak fitting methods examined are listed below and are commonly found in commercial software packages: -

- Average sliding gravity
- Sliding gravity at different thresholds
- Parabolic fit
- Centre of gravity
- Pseudo-Voigt fit.

The methods used vary, but can be categorised as those that fit the peak position using the full peak and those that only use a portion of the peak. For all the materials tested the Pseudo-Voigt fit gave consistently lower scatter. The sliding gravity technique at low thresholds and the average sliding gravity methods also showed reasonable results. Results indicate that in all cases the 80% parabolic fit should be avoided.

The findings indicate that it is preferable to use the whole of the diffraction trace to accurately locate the peak position rather than relying on just a portion of the trace. An example of the degree of scatter between different peak fitting methods is shown in Figure 1, which plots the scatter as a percentage of the mean value for the instrument only repeatability tests.

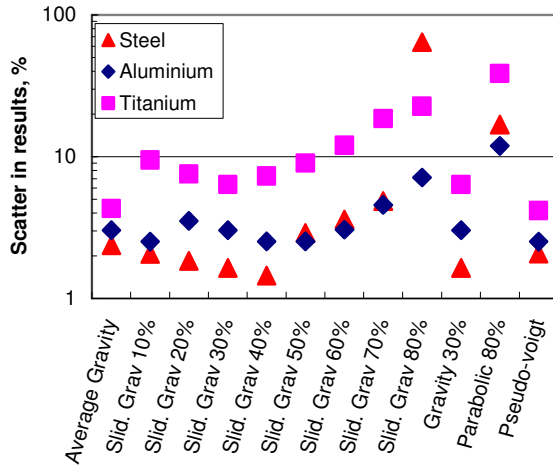


Figure 1 Graph of scatter as a percentage of the mean stress value for different peak fitting routines for the three materials examined.

X-Ray Elastic Constants

Once the diffraction data has been collected, and subsequently analysed such that the peak position has been accurately located, the data can finally be used to calculate the residual stresses present within the test piece. Since the diffraction process measures the strain within the crystal lattice, these measured values need to be converted to stress values using the X-Ray elastic constants (XEC's). Generally most analysis is performed using calculated values from either the Voight or Reuss models, which assume constant strain and constant stress respectively, or an average value obtained from these models can be used, which is known as the Neerfield model.

Issues arise where absolute residual stress values are required from the measurement and not just comparative values from a batch of samples. In this case a greater level of confidence and accuracy is required in the XEC's values. However, presently there are no real points of reference that can be used which provide consistent values for a wide range of engineering materials. The issue therefore is related to what impact the XEC's have on the final calculated stress value. To investigate this a single set of data from the parameter studies was used to examine the impact of a range of values being used in the stress calculation. In this case a set of data from

measurements performed on the shot peened spring steel have been used.

To illustrate the effect the data has been analysed using a range of elastic modulus values, but maintaining the Poisson's ratio and anisotropy constant values the same. If we assume that the true modulus of the steel is 200 GPa, and consider a range of ± 10 GPa ($\pm 5\%$), keeping the Poisson's ratio (ν) at 0.28 and the anisotropy constant (ARX) at 1.49, then the resultant calculated residual stress values cover a range from -509 to -560 MPa. This is shown in Figure 2.

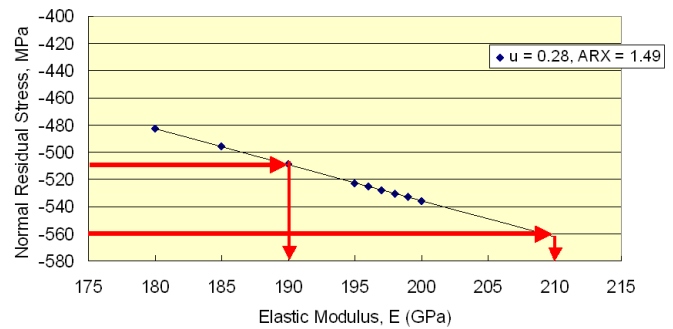


Figure 2 Range of residual stress values obtained from a 10% variance in the assumed elastic modulus.

Since the XEC's values used in stress analysis are products of the elastic modulus and Poisson's ratio, as illustrated in Equation 1 and 2, the combined effect of errors in the elastic modulus and Poisson's ratio, together with an incorrect assumption regarding the specimen anisotropy (where a value of 1 is isotropic and 1.49 is a value recommended by the analysis software for iron based material) significantly increases the potential error in the measurement as shown in Figure 3. The envelope shows the range of values that could be returned from the calculation.

$$-S_1 = \frac{-\nu}{E} \quad (1)$$

$$\frac{1}{2}S_2 = \frac{1+\nu}{E} \quad (2)$$

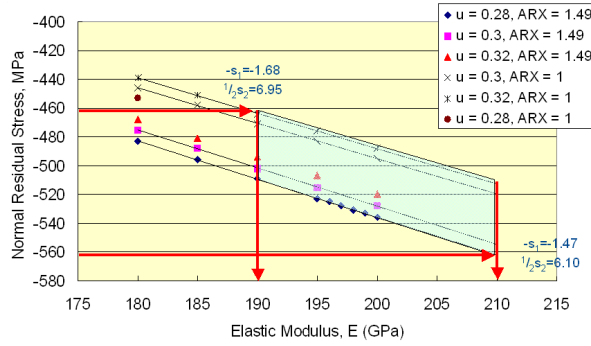


Figure 3 Effect of a $\pm 5\%$ error in the elastic modulus coupled with a $\pm 7\%$ error in Poisson's ratio for a shot peened spring steel sample. The effect of varying the ARX value is also illustrated.

Assuming that the “true” values for the elastic modulus and Poisson's ratio are 200 GPa and 0.3 respectively and that ARX should be 1.49, then an error of $\pm 5\%$ in the elastic modulus (E) and $\pm 7\%$ in the Poisson's ratio (ν) and an incorrect assumption that the specimen is isotropic results in a range of residual stress values as illustrated by the shaded envelope in Figure 3. This results in a residual stress value that ranges from -460 MPa to -560 MPa. That is a possible error of around 19% based on a “true” value of -528 MPa.

The calculated XEC's based on the values of E and ν used in the above example have been included in Figure 3, and are reproduced for clarity below in Table III.

Table III Residual stress values and the XEC's values from the illustration shown in Figure 3.

Elastic Modulus (E), GPa	Poisson's Ration (ν)	ARX	-S ₁	¹ / ₂ S ₂	Residual Stress, MPa
			10 ⁻⁶ MPa ⁻¹		
190	0.32	1	-1.58	6.95	-464
190	0.28	1.49	-1.47	6.74	-509
210	0.32	1	-1.52	6.28	-510
210	0.28	1.49	-1.33	6.09	-560

This shows that the XEC's values vary -1.33 to $-1.58 \times 10^{-6} \text{ MPa}^{-1}$ and 6.09 to $6.95 \times 10^{-6} \text{ MPa}^{-1}$ for $-S_1$ and $\frac{1}{2}S_2$ respectively. Thus giving a range for $-S_1$ and $\frac{1}{2}S_2$ of 0.25 and $86 \times 10^{-6} \text{ MPa}^{-1}$ respectively. Now compare these values in Table IV, which can be found in the literature^[6] for a carbon steel and two crystallographic planes, and which have been calculated from models based on bulk elastic

modulus and Poisson's ratio values, or measured experimentally.

Table IV Experimentally measured, and modelled XEC's for a range of engineering materials from literature^[8]

Method	Value (10^{-6} MPa^{-1})	Steel (% Carbon)	
		0.73	
		(211)	(310)
Mechanical	$-S_1$	-0.94	-0.94
	$\frac{1}{2}S_2$	4.29	4.29
XRD	$-S_1$	-1.02	-1.27
	$\frac{1}{2}S_2$	4.38	5.16
Voight	$-S_1$	-0.85	-0.85
	$\frac{1}{2}S_2$	3.88	3.88
Reuss	$-S_1$	-0.90	-1.57
	$\frac{1}{2}S_2$	4.02	6.04
Neerfield	$-S_1$	-0.88	-1.21
	$\frac{1}{2}S_2$	3.95	4.96

$-S_1$	Max	-1.02	-1.57
	Min	-0.85	-0.85
	Range	0.17	0.72

$\frac{1}{2}S_2$	Max	4.38	6.04
	Min	3.88	3.88
	Range	0.50	2.16

Comparing the range in the XEC's values in the spring steel illustration detailed previously, in which $-S_1$ and $\frac{1}{2}S_2$ had a range of $0.25 \times 10^{-6} \text{ MPa}^{-1}$ and $0.86 \times 10^{-6} \text{ MPa}^{-1}$ respectively, with the values in Table IV, it becomes clear that differences of this order of magnitude in the XEC's obtained from modelling or by direct measurements are not uncommon.

Discussion

When performing XRD residual stress measurements it is clear that the level of precision required will vary depending on the application. For example if the result is required purely for quality control, then provided the same material, test parameters and analysis techniques are used for all measurements then the precise value is not necessary, the customer is merely interested in how one component compares with the other. However, if the exact value is required, for lifetime assessment for example, then it is vital that the most accurate measurement is made. This means that the test set up parameters should be optimised for collecting good quality diffraction data, given the specimen characteristics. It is also

essential that during the data analysis phase the most accurate method of locating the peak position be used. This may not be the same method for all samples and it may be that there are a range of techniques available which yield satisfactory results. Some initial scoping measurements are suggested for new material/stress distributions to establish the reliability of particular peak fitting techniques.

With regards to the XEC's, further work is needed, because, whilst it has been illustrated that there are differences in the values obtained via different methods (i.e. direct measurement or models), it is unclear as to which is the most appropriate and accurate value to use. Ideally the XEC's should be measured for each sample, but this is neither an attractive or realistic proposition, since there are time and material supply implications. What is of importance is that careful consideration is given to the values used in the stress analysis, and that any errors from this value be included in an uncertainty estimate for the measurement.

Conclusion

The scatter in the data is a useful measure for quantifying the effect each parameter has on the uncertainties of the measurement, and this approach has been used to establish guidelines for the NPL Good Practice Guide on the Determination of Residual Stresses by X-Ray Diffraction^[9]. This study has split the XRD stress measurement into two parts, focusing on the test set up and data collection, and the analysis of the data to calculate the stress value. Using three different engineering materials it has been shown that:

- The required degree of accuracy in the stress value depends on the application, for example for quality control the absolute value may not be as important as the variation between batches.
- It has been demonstrated that the measurement technique in itself is self-consistent and gives good repeatability. The repeatability is shown to become worse once there is user intervention.
- With regards to the XRD test set up, providing care is taken, the measurement technique is

reasonably robust. If good counting statistics are obtained then gross positioning errors with regards to the specimen height settings is the most influential factor for introducing errors. Where good counting statistics are not achieved the counting time becomes the most important factor, and longer measurement times are necessary.

- Results also show that the accurate location of the peak position is of great importance, and that not all commercially used peak fitting methods produce a sufficiently accurate value from typical diffraction data. The results presented in this work suggest that it is better to use as much, if not all, of the diffraction trace to accurately locate the peak position.
- Small errors in the values used for the X-Ray Elastic Constants have been shown to have a marked effect on the residual stress value obtained from the analysis. The errors used to illustrate this influence are typical of the range of values that can be obtained from the literature. Care is therefore required when deciding which value to use, and if possible actual measurement of these values^[10] is recommended over the use of calculated values from models.
- Uncertainty budgets can help to focus on areas where there is scope for improvements. Examples of these are presented in the soon to be published second issue of the NPL Good Practice Guide on the Determination of Residual Stresses by X-Ray Diffraction. Additions to this Guide include recommendations regarding depth profiling and data presentation.

References and Further Information

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Additional Information

For further information on residual stress measurement, research projects or to order a copy of any of the NPL reports listed above please contact:

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