Evaluation of the Repeatability of Residual Stress Measurements Using X-Ray Diffraction

Introduction

As part of the CPM4.5 project on the *Measurement of Residual Stress in Components,* a sensitivity evaluation has been carried out to determine the accuracy and uncertainties associated with the X-ray diffraction (XRD) technique for the measurement of residual stresses. Some 250 individual XRD tests have been conducted to evaluate the sensitivity of this technique to changes in the test set-up, and thus quantify the uncertainties inherent in XRD measurements of stress.

This study was not intended to address theoretical issues, which have already been well addressed in the literature, but rather to focus on and evaluate the experimental practicalities of routine XRD stress measurements. A number of key parameters have been examined to evaluate the repeatability of the measurement, the setting up of a sample, the data collection and subsequent data analysis. Measurements were conducted on three materials; a shot peened CCr3 spring steel block, a quenched aluminium block (7010) and a ground piece of titanium alloy. These materials were chosen because they spanned a range of complexities and stress conditions.

This document summarises the key features of the study using the data obtained from the shot-peened spring steel as an example. The full results and findings of this study are available as a separate report, details of which are given at the end of this measurement note.

Tony Fry

May 2002

Measurement Procedure

The three materials were subjected to a similar series of measurements designed to evaluate the relative sensitivity of the XRD technique to changes in the test parameters. The tests were designed to investigate the scatter in the results obtained by introducing deliberate changes in how the test was set-up and conducted.

Scatter in the data is a useful measure for quantifying the effect each parameter has on the accuracy of the measurement, and has been used to establish guidelines for the Good Practice Guide [1], which has been produced as part of this project. The tests have been broken down into two regimes, covering the repeatability of the measurement under ideal conditions and the effect of changes in individual experimental parameters.

Repeatability

To establish the repeatability, two series of tests were carried out on each material. The first was to establish the repeatability of the equipment and data analysis using the system's software. This is referred to as instrument-only repeatability, results shown in Figure 1.

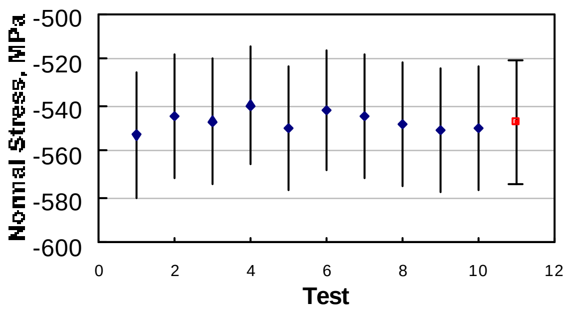


Figure 1. Instrument-only repeatability results showing the 10 individual measurements and the average for the shot-peened spring steel, error bars are from the sin^{2}y fitting

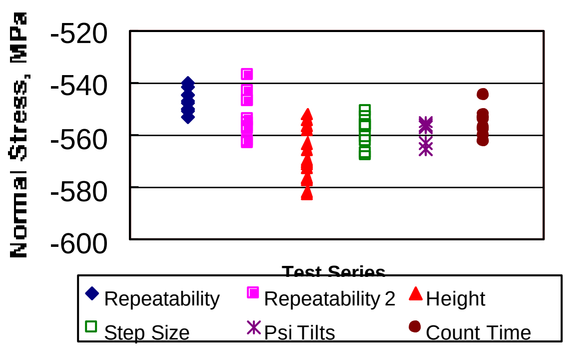
The second was to establish the repeatability in the testing procedure, referred to as instrument-operator repeatability (repeatability 2 in Figure 2). In each case 10 repeat measurements were conducted. In the instrument-only series the specimen was not removed from the equipment, whereas in the instrument-operator series the specimen was removed and replaced between each measurement. In this manner the repeatability of the measurement under normal conditions was established.

Adjustable Parameters

A number of tests were performed on the samples in which one experimental parameter was systematically changed between each subsequent test. In this manner, the effects of the following adjustable parameters were evaluated for their effect on the quality of the measurement.

- Specimen Height Offsets, range ±1mm
- Step Size, over the range 0.01° to 0.1° 2θ
- Number of Psi Tilts, from 5 to 21
- Counting Time
- Peak fitting Techniques

During the tests listed above the sample was not removed from the equipment, thus was no effect caused by operator intervention. A summary plot of the residual stress results obtained from the shot-peened spring steel is presented in Figure 2. The scatter in the results can be expressed as the coefficient of variation (CoV), which is calculated by dividing the standard deviation by the mean



value of the residual stress, these values are presented in Tables I and II.

Figure 2. Summary of results for XRD measurements on the spring steel

Peak Fitting

In conjunction with data collection issues to which the first four parameters listed above pertain, there is a question regarding the data analysis method used. All of the data was corrected for well-documented effects by applying the following series of corrections; *background correction*, *Lorentz-Polarisation* and *Absorption corrections*, Ka_2 *stripping* and *smoothing*. The final stress values were calculated by fitting either a straight line or an ellipse through the sin^2? data points, as appropriate.

As a separate study all of the collected data was analysed using the entire range of peak fitting routines supported by the equipment software used at NPL. These include:

- Sliding gravity (various thresholds[1])
- Average Sliding Gravity
- Centre of gravity (30%)
- Parabolic fit (threshold of 80%)
- Pseudo-Voigt fit

There was a high degree of variation in the results obtained, as shown in Figure 3.

Results

Table I shows the mean values of the residual stress obtained from the parameter evaluation with the associated standard deviation from the data set.

Table I Summary of results for the steel, aluminium and titanium samples

Test Series	Mean Residual Stress, MPa		
	Spring Steel	Aluminium	Titanium
Instrument-Only	-547 ± 4	-198 ± 2	-418 ± 11
Instrument-Operator	-552 ± 8	-202 ± 3	-410 ± 18
Height	-566 ± 10	-212 ± 4	-420 ± 10
Step Size	-559 ± 6	-205 ± 3	-418 ± 11
Psi Tilts	-559 ± 4	-207 ± 2	-427 ± 5
Count Time	-556 ± 5	-206 ± 3	-413 ± 25

The variation in the data sets can be expressed as the coefficient of variation as previously discussed. If the same data is presented in this manner it immediately becomes apparent that the spread in data is not as great as would be initially thought, with the majority of the variation being below 5%, as shown in Table II. The table has been colour coded such that the green cells indicate a CoV between 0 and 1.9%; the amber cells show a CoV of 2.0 to 4.9% and the red cells are for CoV values greater than 5%. The peak fitting study gave a much more alarming variation as shown in Figure 3.

Table II Colour-coded summaries of results for the steel, aluminium and titanium samples

Test Series	Coefficient of Variation, %		
	Spring Steel	Aluminium	Titanium
Instrument-Only	0.7	1.0	2.6
Instrument-Operator	1.4	1.5	4.4
Height	1.8	1.9	2.4
Step Size	1.1	1.5	2.6
Psi Tilts	0.7	1.0	1.2
Count Time	0.9	1.5	5.9

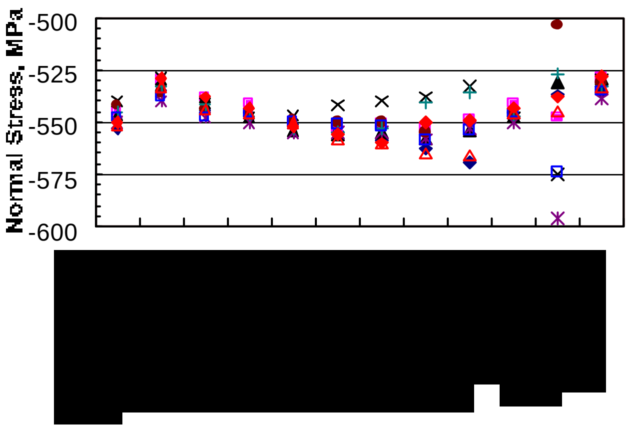


Figure 3. Variation from different peak fitting routines being used on the instrument-only repeatability tests on the shot-peened spring steel

Conclusions

- Generally all the materials tested showed good repeatability in the measurement for both the instrument-only and instrument-operator repeatability tests. Apart from the count time series of tests on the titanium all the results showed less than 5% variance from the mean value.
- It was noted that the repeatability was highly dependent on the peak fitting method used, with the Average Sliding Gravity, Pseudo-Voigt Fit and Sliding Gravity at low thresholds giving the most consistent results with low scatter.
- For guidance on Good Measurement practice the reader is referred to the NPL Measurement

[1] The threshold is defined here as the peak intensity cut off point. Data below this threshold value is not used in the fitting procedure, e.g. for parabolic 80%, only the top 20% of the data is used in the peak fitting routine.

Good Practice Guide No 52: Determination of Residual Stresses by X-ray Diffraction, March 2002.

Further Information

A full report on this work is available. It covers the XRD residual stress measurements in more detail and presents full results on the three materials studied including details of the peak fitting variation. Please ask for an order form from the contact below.

Further information is also available on other aspects of residual stress measurement including:

1 M.E. Fitzpatrick, A.T. Fry, P. Holdway, F.A. Kandil, J. Shackleton and L. Suominen: NPL Good Practice Guide No. 52: Determination of Residual Stresses by X-ray Diffraction. March 2002

2 Sensitivity Evaluation for X-ray diffraction Residual Stress Measurements, NPL Report MATC(A)104, A Fry. April 2002

3 PV Grant, J D Lord and PS Whitehead: NPL Measurement Good Practice Guide No. 53: The Measurement of Residual Stresses by the Incremental Hole Drilling Technique, May 2002

Acknowledgements

This research was carried out as part of project CPM4.5 on the *Methods of Measuring Residual Stress in Components*, which is part of the CPM programme on *Characterisation and Performance of Materials,* funded by the Engineering Industries Directorate of the UK Department of Trade and Industry.

Additional Information

For further information on residual stress measurement, the research project or to order a copy of the full report please contact:

Tony Fry
NPL Materials Centre
National Physical Laboratory
Teddington
Middlesex
TW11 0LW
UK

Telephone:	020 8977 3222 (switchboard)
Direct Line:	020 8943 6220
Facsimile:	020 8943 6722
E-mail:	tony.fry@npl.co.uk

Website: www.npl.co.uk/materials/residualstress