MEASURING RESIDUAL STRESSES IN STAINLESS STEEL - RECENT EXPERIENCES WITHIN A VAMAS EXERCISE

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ABSTRACT

Residual stresses impact on a wide variety of industrial sectors including the automotive, power generation, industrial plant, construction, aerospace, railway and transport industries, and a range of materials manufacturers and processing companies. The X-ray diffraction (XRD) technique is one of the most popular methods for measuring residual stress [1], used routinely in quality control and materials characterisation for validating models and design.

The VAMAS TWA20 Project 3 activity on the “Measurement of Residual Stresses by X-ray Diffraction” was initiated by NPL in 2005 to examine various aspects of the XRD test procedure in support of work aimed at developing an international standard in this area. The purpose of this project was to examine and reduce some of the sources of scatter and uncertainty in the measurement of residual stress by X-ray diffraction on metallic materials, through an international inter-comparison and validation exercise. One of the major issues the inter-comparison highlighted was the problem associated with measuring residual stresses in austenitic stainless steel. The following paper describes this inter-comparison, reviews the results of the exercise and details additional work looking at developing best practice for measuring residual stresses in austenitic stainless steel, for which X-ray measurements are somewhat unreliable and problematic.

INTRODUCTION

The main objective of the VAMAS TWA20 Project 3 was to examine various aspects of the measurement process associated with X-ray residual stress measurement, and contribute to an internationally agreed standard that has been drafted through the CEN/TC138/WG10 committee and the ASTM E28.13 subcommittee on Residual Stress Measurement. The project complimented the activity of CEN group by carrying out additional testing and characterisation in support of the proposed standard, through an inter-comparison exercise. For the inter-comparison a simple component was required that would have a reasonably high stress with good repeatability whilst providing a significant measurement challenge and yet could be produced cost effectively in the numbers required for the first inter-comparison exercise. From previous experience [2, 3] a shot peened stainless steel plate was chosen to meet all these criteria.

The specimens were manufactured from a 316 stainless steel plate, which was cut into 16 large sections and shot peened on one face by Metal Improvement Company, using MI 230 hard media, with an intensity of 0.010 “A and a 200% coverage, where A refers to the Almen measurement, which is a measure of the deflection on a Almen test strip exposed to the peening process.
Eight of the sections were subsequently cut into smaller blocks, approximately 65mm x 50mm x 18mm in size for distribution to the participating laboratories.

Surface roughness measurements were made on the shot peened face of the samples, which was found to have a mean roughness value, $R_a$ of 2.3 µm. Each sample then had reference “baseline” residual stress measurements made at the centre point of each sample using XRD according to the NPL Measurement Good Practice Guide [4]. The results of these measurements are shown in Figure 1.

![Figure 1 Baseline residual stress results for the NPL characterization of the inter-comparison specimens, showing the mean and the 95% uncertainty limits.](image)

The NPL baseline measurements, shown in Figure 1 for all 28 samples, show a larger than expected spread in the values of residual stress. Some inherent variability due to the shot peening process is to be expected, but the scatter in the results may also be due to material effects, since stainless steel is known to be a problematic material to measure using XRD due to the preferred orientation effects. For this reason it was considered to be a challenging material to examine, and therefore a useful extension to previous inter-comparison and validation exercises [2-3]. To reduce the effect of material variability in the inter-comparison, a subset of specimens was chosen from those that fell within the 95% uncertainty limit shown in Figure 1, and these were then circulated to the participants.

To examine the effect of measurement repeatability with the shot peened stainless steel samples, a series of measurements were carried out on a single specimen to compare the uncertainty in the measurements through operator and equipment interaction. The results are presented in Figure 2a. This shows the residual stress measured at the same point repeated ten times with no change to the experimental setup or specimen position. The 95% uncertainty limit is also shown, and for these measurements was calculated to be ±48 MPa.
Further measurements to examine the variation of the residual stress over the surface of a single specimen have also been performed, and are reported in Figure 2b. In this case repeat measurements have been made at different locations on the block, to examine the variability and repeatability of the NPL “baseline” measurements. The measurements were performed at five locations, at the centre of the block and close to each corner. The 95% uncertainty limit is also shown, and for these measurements was calculated to be ±44 MPa. This is lower than the value calculated for the series of repeat measurements at the same location (and indicates that the stress variation across the surface of a single sample is likely to contribute less to the overall uncertainty than the inherent uncertainty associated with repeat measurements.

**INTER-COMPARISON RESULTS**

Over 20 laboratories agreed to take part in the inter-comparison with results finally being received from 13 of these. The results presented in this paper have been received from the UK, Germany, Finland, USA, Canada and Japan, representing the major countries involved in the VAMAS organisation. The participants were asked to perform residual stress measurements on the center of the shot peened surface. They were also asked to perform repeat measurements so that the repeatability of their equipment and measurement could be assessed. Participants were allowed to use their own in-house measurement procedure, and asked to report the details regarding the equipment used and the procedure followed when reporting their results. A breakdown of the range of equipment used and key experimental variables are presented in Figure 3. This shows that the measurements were predominantly made using commercial laboratory based diffractometers, with a fairly even split between Cr-kα and Mn-kα radiation sources. The majority of participants used area type detectors such as position sensitive detectors, whilst a variety of peak location methods were used.

The results of the measurements made by all participants are presented in Figure 4. The repeat measurements on the sample are shown in Figure 4. The uncertainty in the measurements has then been calculated according to the NPL Good Practice Guide [4] and is shown by the error bars on the mean measurement values (triangle symbols). Generally there was good agreement between the NPL reference data and participants.
results, although it should be noted that some of the repeatability measurements did show a large amount of variability, in one case as much as 200 MPa. Figure 4 also shows that the results fall into two distinct bands, giving average stress values of approximately -450 MPa or -650 MPa respectively. From further examination of the returned data an explanation of the formation of these two bands can be found.

![Figure 3 Breakdown of the type of equipment and key experimental parameters used by the participants of the inter-comparison.](image)

**Figure 3** Breakdown of the type of equipment and key experimental parameters used by the participants of the inter-comparison.

![Figure 4 Residual stress measurements and X-ray source used by the participants compared to the NPL reference measurements, and their associated uncertainties.](image)

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Figure 4 also shows the X-ray source used by the participants, namely Cr-\(\alpha\) or Mn-\(\alpha\). Whilst both of these sources are recommended in the literature [4,7,8] as suitable sources for measurement of stainless steel, consideration of the different sources used could account for the banding of the results, but there are some discrepancies that require further explanation. For instance there are two clear occurrences of measurements not following this trend. The first is with Cr radiation producing highly compressive results,
as seen with the fourth data set in Figure 4, the second being the sixth data set that uses Mn radiation but has lower compressive stresses than this explanation would suggest.

The difference in the results obtained from the two different X-ray sources is not wholly unexpected. The penetration depth for Cr-\( \kappa \alpha \) and Mn-\( \kappa \alpha \) X-rays is different — approximately 10 and 16 \( \mu \text{m} \) respectively in austenitic stainless. With the stress gradient associated with the shot peened surface, a difference in the measured stress would be expected. In addition the different X-rays are diffracting from different crystallographic planes, hence this too may account for some of the differences. Incremental hole drilling measurements on these specimens has shown that given the different penetration depths of the two X-ray sources and the stress gradient in the shot peened samples a difference in the measured stress value of around 50 MPa might be expected, but this does not fully explain the \( \sim 200 \) MPa difference seen in Figure 4. Previous studies comparing incremental hole drilling and XRD [5] have shown the two techniques to generally agree well. In this case the hole drilling measurements were \(-550\) MPa compared to the reference XRD measurement of \(-425\) MPa. This could be due to the surface roughness of the specimens and/or variation of stress over the surface of the specimens, which has been shown to be in the order of \( \pm 50 \) MPa. Whilst the agreement of the magnitude of the near surface values can be debated, the gradient and penetration depths still cannot fully explain the differences in the results. Another factor that may influence the residual stress measurement is the method of data analysis. Previous studies of the XRD method for residual stress measurements have shown that the method of peak location can have a profound effect on the accuracy and repeatability of the measurement [6], as shown in Figure 5. This figure shows repeat measurements made on a spring steel block that had been shot peened, and results analysed using a range of peak location methods. The data shows the measurements are critically dependant on the analysis method used. Techniques which use the whole of the diffraction data have been shown to be more reliable than techniques which only use a portion of the diffraction data, such as the parabolic fit function which has a 100 MPa spread in Figure 5 as compared to the Pseudo-voigt fit which has a \( \sim 15 \) MPa spread.

![Figure 5 Typical repeatability results for shot peened spring steel, analysed with different peak fitting methods.](image-url)
By overlaying the peak fitting method onto the data shown in Figure 4, it is possible to identify additional contributions to the banding of the results, as shown in Figure 6. This shows that the results in the upper band, i.e. those that are in best agreement with the reference measurements, were analysed using techniques that use the whole diffraction data. The results that form the lower band and differ most from the NPL ‘reference’ results have been obtained using peak fitting methods that use only a portion of the diffraction data. Figure 5 shows a potential 100 MPa spread in stress values when using the parabolic peak fitting method, which is similar in form to the Gaussian and FWHM methods. Also, the different penetration depths of the two X-ray sources used can lead to an additional 50 MPa compressive stress in the results obtained using the Mn-\(\kappa\alpha\) source. When both these factors are taken into account, it is possible to adjust the suspect results so that they fall into one scatter band, as shown in Figure 7.

![Figure 6](image1.png)

**Figure 6** Participant’s results, showing the peak fitting method used.

![Figure 7](image2.png)

**Figure 7** Schematic showing how the data could fall into one scatter band by ‘correcting’ for penetration depths and peak fitting techniques.
Additional measurements conducted at NPL using both Cr-\(k\alpha\) and Mn-\(k\alpha\) radiation. Stress measurements were made using a Bruker D8 Discover diffractometer under iso-inclination conditions, using 9 psi tilts in both the positive and negative directions. Measurements were made in accordance with the recommendations in Ref 4, and the analysis performed using an elastic modulus of 195 GPa and a Poisson’s ratio of 0.3, as prescribed in the inter-comparison instructions. The resultant diffraction peaks were analysed using three peak fitting techniques; a pseudo-voigt fit, a parabolic fit and the centre of gravity method. The results of these measurements are presented in Figure 8, which shows the uncorrected inter-comparison data and the new NPL data. This shows that good agreement can be obtained from data generated using both X-ray sources, and supports the results shown in data sets 4 and 5 of Figure 4. The results also show the effect on the reported stress magnitudes and clearly illustrate how the two bands could have been formed.

![Figure 8 Inter-comparison results and additional measurements performed at NPL using Mn-\(k\alpha\) and Cr-\(k\alpha\) radiation and analysed using three peak fitting techniques, error bars for the additional measurements are included.](image)

DISCUSSION AND CONCLUSION

An inter-comparison exercise run under the auspices of VAMAS TWA20 Project 3 produced some disconcerting residual stress measurements. It has been demonstrated that an initially problematic data set from an international inter-comparison exercise can be rationalised. It has been demonstrated that the discrepancies were not due to the choice of X-ray source, but to the analysis method used in the treatment of the X-ray data.
Through further measurements using both X-ray sources and a range of peak fitting methods on data from one specimen, similar degrees of scatter in the results to those reported by the participants of the exercise have been obtained. This clearly shows that laboratories need to consider carefully which peak fitting technique they use in residual stress measurement, as it is possible to obtain a wide range of results from the same data set. Whilst it is recognised that with some materials and stress states, collection of the complete diffraction peak, back to background levels, is not possible (where area detectors are used for example), it is recommended that users should wherever possible do this. It is also recommended that the diffraction data should be analysed using methods that use the complete diffraction data set, as these are the preferred techniques since they have consistently shown to improve reliability and repeatability in the analysis of XRD data.

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REFERENCES