

# **Residual Stress Measurement: XRD Depth Profiling Using Successive Material Removal**

## **Introduction**

One of the most popular non-destructive methods for measuring surface residual stresses<sup>[1]</sup> is X-ray diffraction (XRD). The scope of this technique can be increased by using XRD in combination with electro-polishing and successive incremental material removal to obtain information on the variation of residual stress with depth. Using XRD in such a 'semi-destructive' manner provides a measurement technique that furnishes data, which has traditionally been obtained using neutron diffraction, synchrotron diffraction and hole drilling methods. Such an approach can be defined as 'semi-destructive' since although material has been removed, it is possible to perform repeat measurements at every incremental step, unlike the 'destructive' hole drilling technique.

Generally however no allowance is made for any redistribution of stress that may have occurred as part of the material removal process, but this can have significant effect on the values measured.

As part of a continuing effort to improve and refine residual stress measurement using the XRD technique, NPL has recently completed a series of measurements on a nickel based superalloy, which had been homogeneously shot-peened on both sides by Metal Improvement Co., UK using parameters representative of commercial practice. The purpose of these measurements was to investigate and illustrate how progressive material removal can significantly relax the stresses generated by peening.

In this investigation XRD measurements conducted at NPL are used in conjunction with neutron diffraction measurements performed by Imperial College on ENGIN at ISIS, Rutherford, UK.

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## Introduction

A three year in-depth study of residual stress measurement techniques recently conducted at NPL has shown that residual stress measurements made just below the surface by X-ray diffraction can be both highly repeatable and reproducible. As part of this study a Good Practice Guide <sup>[2]</sup> has been written which offers advice and recommendations for the successful completion of near surface residual stress measurements. As a continuation of this study, a new project will seek to further refine the measurements technique and expand on the Good Practice Guide. This will include a review of the literature for recommended electro-polishing conditions for common engineering materials and an examination of the methods available for measuring how the residual stress present within a material varies with depth.

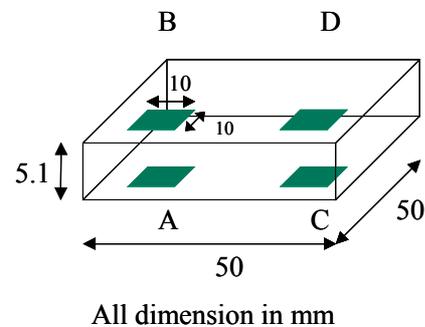
Preliminary literature searches have indicated that electro-polishing conditions are not always reported and that a collection of recommended 'recipes' and conditions for commonly used materials, coupled with a guide for frequently encountered problems and possible solutions to these problems, would be beneficial to the users of the technique. Table I presents some initial examples of common problems encountered when electro-polishing together with the probable causes and solutions.

Initial studies have been conducted as part of the VAMAS TWA20 activity <sup>[3]</sup> to develop a Code of Practice for making reliable residual stress measurements by neutron diffraction. As part of the current work raw and corrected XRD data have been compared with the neutron diffraction data which were made at various depths through the material and in regions which had been electro-polished, so that the effect of material relaxation could be assessed.

## Material

Inconel 718, a nickel based superalloy, was used for this study. The specimen which was 50 mm square and 5.1 mm thick was removed from an aero engine disc forging by means of a milling cutter. In general the maximum grain size was reported to be ASTM 6 or finer, however grains as

large as ASTM 3 were reported to be occasionally present. The upper and lower sides were then homogeneously shot-peened by Metal Improvement Co., UK, using shot-peening parameters that were representative of a commercial treatment, a shot size MI 230R, intensity 0.0083 A with a coverage of 200%. Four regions, two on each face and 10 mm<sup>2</sup>, were identified on the specimen and assigned the identifiers A, B, C and D. Three of these square regions were then electro-polished with depths of material removal of 70 μm (B), 130 μm (C) and 190 μm (D). These are shown schematically in Figure 1. Position A was left un-polished in the as-received state.



**Figure 1 Schematic diagram of the IN718 specimen showing the measurement locations A, B, C and D.**

## X-ray Diffraction

X-ray diffraction residual stress measurements were performed using a Siemens D500 diffractometer set up in the Bragg-Brentano geometry and using Cr-K $\alpha$  radiation. The residual stress was measured in three orientations, X, Y and XY (at 45°). A series of 7 repeat measurements were performed without removing the specimen between successive measurements. The residual stress measurements were performed in accordance with the NPL Measurement Good Practice Guide No. 52 – Determination of Residual Stresses by X-ray Diffraction <sup>[2]</sup>. The magnitude of the residual stress was evaluated using the Bruker STRESS<sup>plus</sup> program. The penetration depth of the X-ray for 95% of the diffracted signal was calculated to be 8.5 μm.

**Table I Common problems that occur when performing electrolytic polishing and some suggested remedies commonly cited in the literature.**

| <b>Problem</b>  | <b>Probable cause</b>   | <b>Suggestions</b>  |
|---|---|---|
| The centre of the polished region is too deeply attacked.                   | The polishing film did not form in the centre of the desired polished area.     | <ul style="list-style-type: none"> <li>• Increase the voltage of the electro-polisher.</li> <li>• Reduce the amount of agitation.</li> <li>• Use a higher viscosity electrolyte.</li> </ul>   |
| Pitting and attack at the edge of the specimen.                             | The polishing film is too viscous or it is too thin.                            | <ul style="list-style-type: none"> <li>• Decrease the voltage of the electro-polisher.</li> <li>• Increase the amount of agitation.</li> <li>• Use a less viscous electrolyte.</li> </ul>   |
| Deposits are formed on the surface of the specimen.                         | The products formed at the anode are likely to be insoluble.                    | <ul style="list-style-type: none"> <li>• Choose a different electrolyte which will not form insoluble products.</li> <li>• Raise the temperature of the system.</li> <li>• Increase the voltage of the electro-polisher.</li> </ul> |
| The surface of the specimen is rough or has no lustre.                      | The polishing film is likely to be inadequate.                                  | <ul style="list-style-type: none"> <li>• Increase the voltage of the electro-polisher.</li> <li>• Try using a more viscous electrolyte.</li> </ul>  |
| There are undulations or scratches on the polished surface of the specimen. | Incorrect polishing time.<br>Inadequate agitation.<br>Unsuitable preparation.   | <ul style="list-style-type: none"> <li>• Try altering the amount of agitation.</li> <li>• Improve the preparation procedure.</li> <li>• Increase the voltage of the electro-polisher and decrease the polishing time.</li> </ul>    |
| There are stains on the polished surface.                                   | Etching has occurred after the current has been switched off.                   | <ul style="list-style-type: none"> <li>• Remove the specimen immediately after the current is switched off.</li> <li>• Choose a less active electrolyte.</li> </ul>   |
| Regions of the surface have not been polished.                              | There are likely to have been gas bubbles masking the surface.                  | <ul style="list-style-type: none"> <li>• Increase the amount of agitation.</li> <li>• Decrease the voltage of the electro-polisher.</li> </ul>  |
| Different phases are in relief.   | The polishing film is likely to have been inadequate.                           | <ul style="list-style-type: none"> <li>• Increase the voltage of the electro-polisher.</li> <li>• Improve the preparation procedure used.</li> <li>• Reduce the polishing time.</li> </ul>  |
| Pitting.  | Polishing time is too long.<br>The voltage of the electro-polisher is too high. | <ul style="list-style-type: none"> <li>• Improve the preparation procedure used.</li> <li>• Decrease the polishing time used.</li> <li>• Try various electrolytes.</li> </ul>   |

**Neutron Diffraction**

The neutron diffraction measurements were carried out as part of VAMAS TWA20 on ENGIN at ISIS, Rutherford, UK. This is a time of flight instrument, which is capable of detecting multiple diffraction peaks. These are then refined using a Rietveld refinement, which involves fitting a crystallographic model of the structure to the entire diffraction spectrum. This provides the value for the strained lattice parameter which can then be used to calculate the residual stress. For these measurements a sampling volume of 0.5 mm in height by 5 mm x 1.4 mm was used. These dimension helped to ensure good resolution perpendicular to the specimen surface. An elastic modulus of 200 GPa and a Poisson’s ratio of 0.3 were used for both the XRD and neutron stress

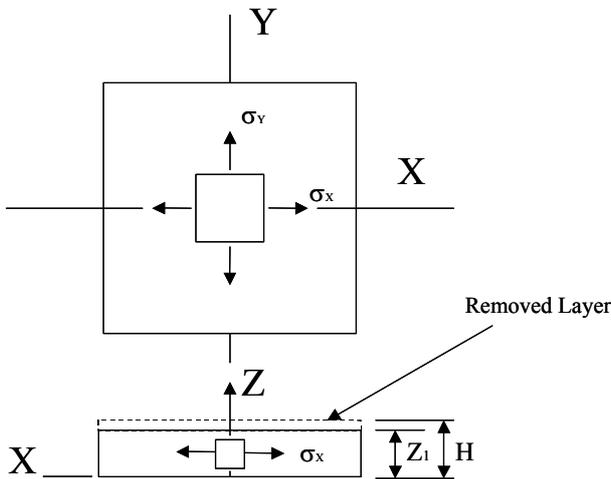


Figure 2 Stresses in a flat plate after layer removal

calculations.

**Electro-polishing Correction**

Measuring residual stresses as a function of depth with X-rays requires the removal of material. Any mechanical method of removal, no matter how fine the abrasive or machining method will deform the surface and introduce additional stresses, thereby altering the stress state that was originally present in the material. Such methods should therefore be avoided. Electro-polishing is the preferred method for removing material for the purposes of residual stress depth profiling, although a combination of machining or grinding followed by electro-

polishing has been reported to be suitable for the removal of thick layers [4]. When performing layer removal for residual stress depth profiling it is important to consider any redistribution or relaxation in the residual stress in the exposed surface, particularly if the component is relatively thin. Solutions are available to correct the stress values obtained. For a flat plate (Figure 2) a generalized solution proposed by Sikarskie [5] based on the original solutions of Moore and Evans [6] can be used. This takes the proposed solutions and expands the integrands in a Taylor’s series, as shown below starting from the generalized solution:

$$\sigma(z_1) = \sigma_m(z_1) + 2 \int_{z_1}^H \frac{\sigma_m(z)}{z} dz - 6z_1 \int_{z_1}^H \frac{\sigma_m(z)}{z^2} dz$$

Expanding the above integrands using a Taylor’s series results in the following equation:

$$\sigma(z_1) = \sigma_m(z_1) + \left( -4\sigma_m(H) \left( \frac{H-Z_1}{H} \right) + [\sigma_m(H) + 2H\sigma'_m(H)] \left( \frac{H-Z_1}{H} \right)^2 + \frac{1}{3} [2\sigma_m(H) + H\sigma'_m(H) - 2H^2\sigma''_m(H)] \times \left( \frac{H-Z_1}{H} \right)^3 \dots \right)$$

This can be further simplified by performing electrolytic polishing for shallow depth increments (a few percent of the specimen thickness). In this case only the first term of the series is necessary for the correction of the raw data:

$$\sigma(z_1) = \sigma_m(z_1) + \left( -4\sigma_m(H) \left( \frac{\Delta Z_1}{H} \right) \right)$$

where H is the original plate thickness,  $\Delta Z_1$  is the change in thickness after layer removal,  $\sigma_m$  is the measured stress and  $\sigma_z$  is the corrected stress.

**Results**

The X-ray diffraction measurements are shown in Table II. The depth at which the measurement is quoted is the sum of the polishing depth and the calculated penetration depth of the X-ray beam. For example for the measurements made at location A, the effective depth of the measurement, d can be expressed as:

d = material removed + penetration depth,

$$d = 0 + 0.0085 \text{ mm}$$

The raw data and corrected values at each location are given in Table II with the uncertainty that has been evaluated in accordance with the NPL Good Practice Guide No. 52 [2].

**Table II XRD Residual stress values**

| Location and depth (mm) | Residual Stress, MPa |           |             |
|-------------------------|----------------------|-----------|-------------|
|                         | Raw                  | Corrected | Uncertainty |
| A: 0.0085               | -631                 | -631      | ±55         |
| B: 0.0765               | -877                 | -844      | ±72         |
| C: 0.1365               | -530                 | -467      | ±46         |
| D: 0.1985               | -220                 | -126      | ±23         |

The neutron diffraction measurements are shown in Table III. The data in the first column have been obtained by scanning through the material to different depths below the unpolished region A, this means that this data has not been influenced by any possible stress redistribution which may have resulted from the electro-polishing process. Further identical neutron diffraction residual stress measurements were made at locations B, C and D, as reported by Bonner *et al* [3]. These values are also presented in Table III, and show some difference in the residual stress values measured at the equivalent depth as those made at position A.

**Table III Residual stress values from neutron diffraction measurements**

| Location and depth (mm) | Residual Stress, MPa |      |      |     |
|-------------------------|----------------------|------|------|-----|
|                         | A                    | B    | C    | D   |
| A: 0.0528               | -920.5               |      |      |     |
| B: 0.1312               | -642                 | -646 |      |     |
| C: 0.1636               | -378.5               |      | -369 |     |
| D: 0.2329               | -54                  |      |      | -31 |

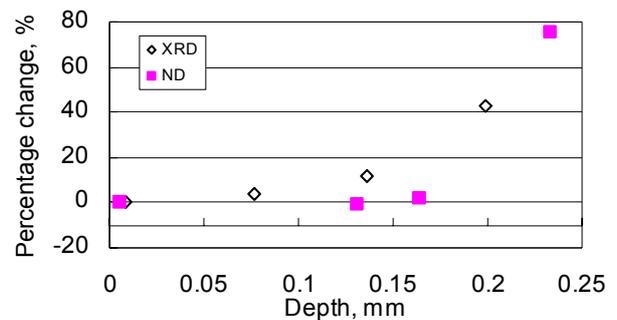
In this case, with a relatively thick specimen geometry, the results show that stress redistribution appears to be more pronounced at greater depths of material removal. This is further illustrated in Figure 3, which shows the change in the residual stress values expressed as percentages

using the following methods for the XRD and the neutron diffraction data:

XRD – the percentage change in this case is the difference between the ‘raw’ and the ‘corrected’ data expressed as a percentage of the original ‘raw’ data value.

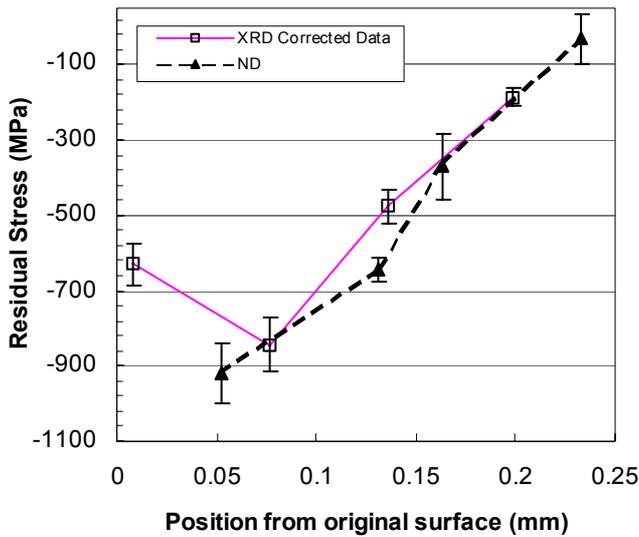
ND – the percentage change in this case is the difference between the data measured at location A and the values measured at the electro-polished locations B, C and D. This difference is expressed as a percentage of the value measured at location A.

Figure 3 shows that for shallow depth increments of material removal the percentage change in the residual stress values for the XRD and neutron methods are of a similar magnitude, around 5 to 10%. With increasing depth the residual stresses approach zero (this occurs at around 0.25 mm from Figure 4), so any error in the measured stress value caused by redistribution has a more significant effect on the measurement accuracy and uncertainty. The last data point in Figure 3 illustrates this and consequently is somewhat misleading. Since there were no XRD measurements conducted at this depth it is unclear as to the effect on the XRD data.



**Figure 3 Percentage change in the residual stress value caused by corrections (XRD) or stress redistribution (ND)**

The results are plotted against depth in Figure 4. This Figure presents the corrected XRD data plotted for comparison with the neutron diffraction data and associated error measured at the four electro-polished locations.



**Figure 4 Comparison of XRD and ND (Pos A, B, C and D) near surface stress measurements on shot peened nickel based superalloy (IN718)**

**Discussion**

Figure 4 shows the XRD data to be in good agreement with the neutron diffraction data. This illustrates that by using electro-polishing techniques to remove shallow depths of material, localised stress relaxation can be minimised, thus producing accurate and reliable residual stress measurements using X-ray diffraction. Comparing the corrected XRD data with the neutron diffraction data that was measured near the surface of the electro-polished regions B, C and D (as shown in Table III) as opposed to purely below region A, further illustrates the requirement to correct XRD data.

Although the correction seems minor given the magnitude of the stresses, it does provide improved measurement accuracy. Interestingly, apart from the deviation in the neutron data at a depth of 0.1312 mm there is linear trend in the stress profile as material is removed, which is also evident in the XRD data. In fact had this deviation not occurred the data points would be more or less co-linear, as illustrated in Figure 4.

**Conclusions**

- Electrolytic polishing is the recommended method for material removal for the purposes of

residual stress depth profile measurement using X-ray diffraction.

- Through comparison it has been shown that residual stress measurements obtained using X-ray diffraction techniques are in good agreement with results obtained by neutron diffraction.
- It has also been illustrated that XRD can be corrected for the material relaxation caused by the electrolytic polishing, as reported by Bonner *et al* [3], further comparison of this corrected data with neutron measurements has shown improved agreement.
- It has been illustrated that the use of a simplified general solution for flat plates can be used for shallow depths of material removal; in this case of the order of 4-5% of the plate thickness.
- Solutions for correcting the measured residual stress values are well documented within the literature and exist for different specimen geometries. The reader is referred to references 5-8 for further information.
- During the course of this work, information relating to electrolytic polishing was sought within the literature. Whilst commonly used, the author found very little reported conditions or recommendations. Further work and exploration of the open literature is therefore required to establish best practice in terms of X-ray diffraction residual stress depth profiling. These and other issues will be addressed within the MPP8.5 project.
- For further guidance on good XRD measurement practice the reader is referred to the NPL Measurement Good Practice Guide No 52: Determination of Residual Stresses by X-ray Diffraction, March 2002.

## 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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