Residual Stress Measurement: XRD Depth Profiling Using Successive Material Removal

Summary

The variation of residual stress with depth has been measured using XRD and compared with other established techniques such as incremental hole drilling and neutron diffraction. Details of the measurements conducted on a number of specimens with different geometries and processing histories are presented and issues relevant to depth profiling and the corrections necessary for strain relaxation for these geometries are discussed.

Introduction

One of the most popular non-destructive methods for measuring residual stresses [1] is X-Ray diffraction (XRD). With a typical penetration depth of around 10 to 20 μm the measurements are essentially near surface, but by using XRD in combination with electro-polishing and successive incremental material removal the scope of this technique can be increased such that information on the variation of residual stress with depth can be obtained. 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.

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 materials that have been processed in different ways to establish the reliability of XRD depth profiling. In this study XRD and fine incremental hole drilling measurements conducted at NPL are compared in conjunction with neutron diffraction measurements performed by Imperial College on ENGIN-X at ISIS, Rutherford, UK.

During the material removal process there will be some stress redistribution. Since this can have a significant effect on the values measured, corrections are required. Examples of the form of these corrections are presented.

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September 2005

Introduction

Over the last six years as part of two DTI funded projects NPL has been performing an in-depth study of residual stress measurement. This has focused on the more popular techniques such as X-Ray diffraction and hole drilling, but also included neutron and synchrotron diffraction and the emerging methods such as MAPS and the contour method. As part of this study a Good Practice Guide [2] has been produced which offers advice and recommendations for the successful completion of near surface residual stress measurements using XRD.

This guide has recently been extended to include recommendations for depth profiling. As part of this effort, users of the depth profiling technique were surveyed to establish the experimental methods commonly used. In addition a number of comparison measurements have been conducted on a range of materials that had different surface treatments. The following measurement note summarises the responses from industry to the questionnaire and gives further details of several of the comparison measurements conducted as part of these projects.

Survey of Industry

A questionnaire was distributed amongst key UK X-Ray diffraction users, to establish the range of equipment and the experimental techniques used. The results from the questionnaire are summarised as follows.

Of the replies received most laboratories used commercial polishing equipment. Polishing depths ranged from 10 microns to 1 mm with areas of material removal from 50 mm^2 to 175 mm^2. Three main methods were used for measuring the amount of material removed (i.e. the new thickness of the sample) after polishing, which were in order of popularity; micrometer, dial gauge and by use of a calibrated microscope.

The range of materials on which depth profiling was routinely performed covered typical engineering materials, steel, austenitic stainless steel, aluminium, titanium, inconel, zirconium and nickel alloys.

Depth profiling comparison measurements

Knowledge of the variation of residual stress with depth can be of great importance in certain loading conditions, e.g. for crack initiation and growth studies and to evaluate the effectiveness of different surface treatments. Hence it is vital to have the ability to accurately measure this. This type of near surface measurement (approximately to depths of 1 mm) can be most readily performed using XRD or hole drilling methods. Historically the hole drilling method was performed using a single depth measurement or several large ($\approx$128 μm) increments. Work at NPL has focused on a procedure for fine incremental hole drilling (with depth increments as small as 10 μm) which provides greater detail in the near surface region than previously possible. By way of validating this data, comparison measurements have been made with XRD,

1. By comparison with Neutron diffraction data to establish confidence in the XRD method.

2. Validation of the hole drilling data by comparison with XRD data for:

   - different shot peening treatments, and
   - wet and dry machining.

XRD Comparison with Neutron Diffraction

To be confident with future comparisons with hole drilling measurements, initial studies were performed comparing XRD data and neutron diffraction data on the same sample to establish and verify the reliability of the XRD technique. These measurements were conducted as part of the VAMAS TWA20 activity [3] to develop a Code of Practice [4] for making reliable residual stress measurements by neutron diffraction.

Raw and corrected XRD measurements were compared with the neutron diffraction data, which were made at various depths through the material and in separate regions which had been electro-polished to different depths, so that the effect of material relaxation could be assessed.

The material used for this exercise was Inconel 718, a nickel based superalloy. The specimen, which was 50 mm x 50 mm x 5.1 mm thick was removed from an aero engine disc forging by means of a milling cutter. The upper and lower sides were then homogeneously shot-peened by Metal Improvement Co., UK, using processing parameters that were representative of a commercial treatment. Four regions, two on each face and 10 mm^2, were identified on the specimen and assigned the identifiers A, B, C and D. Three of these 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.

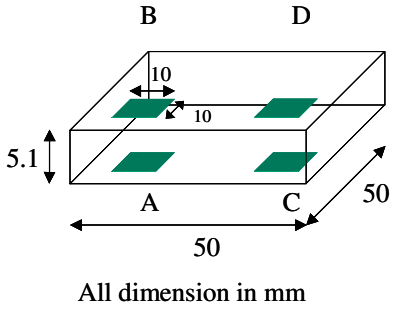

Figure 1 Schematic diagram of the IN718 specimen showing the measurement locations A, B, C and D.

X-Ray Diffraction

XRD residual stress measurements were performed at NPL using a Siemens D500 diffractometer set up in the Bragg-Brentano geometry and using Cr-Kα 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 [2]. The magnitude of the residual stress was evaluated using the Bruker STRESS^{plus} program.

Neutron Diffraction

The neutron diffraction measurements were carried out as part of the VAMAS TWA20 study on ENGIN-X 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. For these measurements a sampling volume of 0.5 mm in height by 5 mm x 1.4 mm was used, which 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 calculations.

Electro-polishing Correction

When performing layer removal for residual stress depth profiling it is important to consider the potential for any redistribution or relaxation in the residual stress in the exposed surface, particularly if the component is relatively thin. Various solutions are available to correct the stress values obtained [5-6]. For a flat plate (Figure 2) a generalized solution proposed by Sikarskie [5] based on the original work of Moore and Evans [6] can be used.

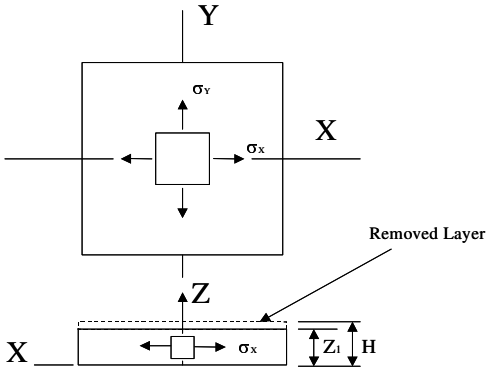

Figure 2 Stresses in a flat plate after layer

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:

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

This can be simplified if electrolytic polishing is carried out over 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, thus:

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

where H is the original plate thickness, ΔZ_1 is the change in thickness after layer removal, σ_m is the measured stress and σ_z is the corrected stress.

In the case of hollow cylindrical sample with rotational symmetric stresses, as shown in Figure 3, the following corrections can be applied.

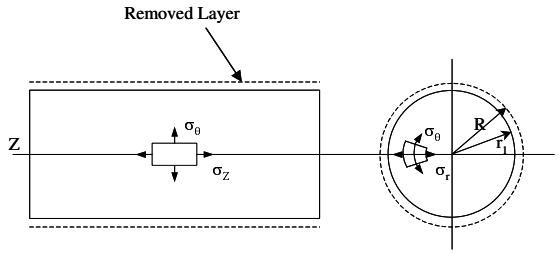

Figure 3 Rotationally symmetric stresses in a cylinder.

$$\sigma_r(r_1) = -\left(1 - \frac{R_1^2}{r_1^2}\right)\int_{r1}^{R}\left(\frac{r^2}{r^2 - R_1^2}\right)\frac{\sigma_{\theta m}(r)}{r}dr$$

$$\sigma_z(r_1) = \sigma_{zm}(r_1) - 2\int_{r_1}^{R}\left(\frac{r^2}{r^2 - R_1^2}\right)\frac{\sigma_{zm}(r)}{r}dr$$

$$\sigma_\theta(r_1) = \sigma_{\theta m}(r_1) + \left(\frac{r_1^2 + R_1^2}{r_1^2 - R_1^2}\right)\sigma_r(r_1)$$

where R is the original radius of the cylinder, and r_1 is the radius at the depth of interest.

Results

The XRD measurements are shown in Table I. 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.

The raw data and corrected values at each location are given in Table I with the associated uncertainty that has been evaluated in accordance with the NPL Good Practice Guide No. 52 [2]. This is based on the repeatability of the measurement and the error reported by the analysis software.

Table I 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 II. 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 II, and show some difference in the residual stress values measured at the equivalent depth compared to those made at position A.

Table II 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

It should be noted that the effective depths are different in Tables I and II, but both sets of 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. This shows the XRD data to be in good agreement with the neutron diffraction data. Figure 4 also illustrates the benefit of XRD

over neutron diffraction in the very near surface regions of around 10 to 20 µm.

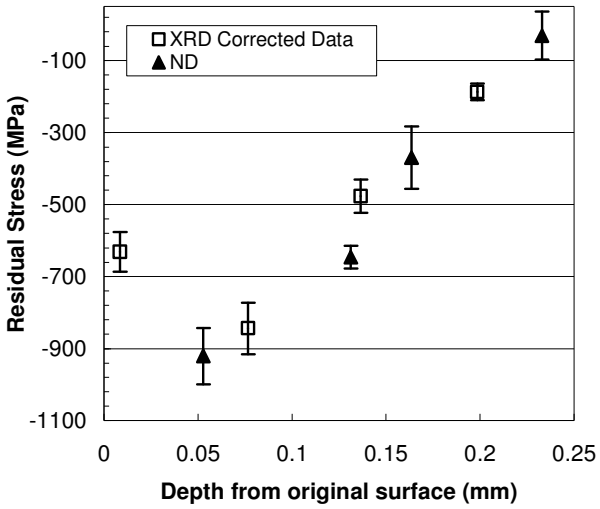

Figure 4 Comparison of XRD and ND (Pos A, B, C and D) near surface stress measurements on shot peened nickel based superalloy (IN718)

XRD Comparison with Hole Drilling

Having established the reliability of the XRD technique against neutron diffraction data, further XRD measurements were made on a number of materials with different surface preparations. These measurements have been compared with fine increment hole drilling measurements to help validate this technique.

Shot Peened Aluminium Plate

Measurements were carried out, using the D500 diffractometer previously described, on a lightly peened 7000 series Al sample. Successive measurements at approximately 25 µm intervals were performed and compared with conventional and fine hole drilling measurements (see Figure 5). Electro-polishing was conducted using a Struers Electrolpol 5 polisher in conjunction with an electrolyte consisting of Ethanol 64-17-5 (60-100%), 2-Butoxyethanol 111-76-2 (10-15% 0, Perchloric acid 60% 7601-90-3 (1-5%).

As before the XRD data has been corrected using the flat plate correction detailed previously. The penetration depth of the Cr Ka X-Ray beam has been taken as the first measurement depth. Apart from a very slight offset close to the surface of the specimen, the data sets agree very well. The offset may be due to the zero point detection in the hole drilling measurements. Also note how Figure 5 clearly demonstrates the advantage of fine increment hole drilling over the conventional (128 µm increment) method, where the detail of the peak compressive residual stress would have been lost using the conventional method.

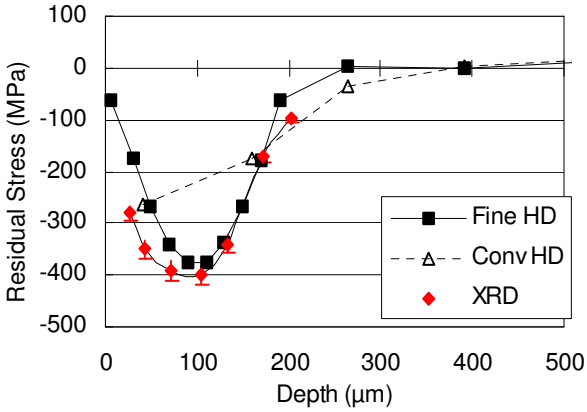

Figure 5 Comparison of residual stress profiles measured using XRD and fine and conventional increment hole drilling.

Wet and Dry Machining

The influence of different machining conditions on near surface residual stresses has also been examined on a set of 321 stainless steel discs subjected to a wet and dry turning operation. Since the stress distributions generated from the two conditions were very similar, only the dry machining data is presented in Figure 6, which once again shows good agreement between the two methods. The circumferential (hoop) stresses, as defined in Figure 7, for the dry machined discs were approximately 38% higher than those generated during wet machining, but there was little difference in the radial stresses for the two conditions.

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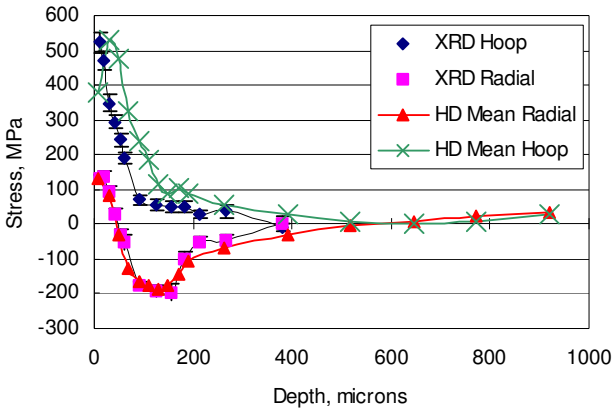

Figure 6 XRD and hole drilling data for a dry machined 321 stainless steel disc.

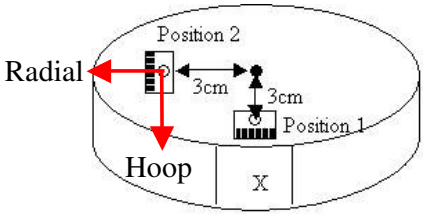

Figure 7 Schematic of the dry machined disc showing the XRD measurement directions and hole drilling locations.

Conclusions

- Electrolytic polishing is the recommended method for material removal for residual stress depth profile measurements using X-Ray diffraction. If large depth increments are required it is possible to combine electro-polishing with conventional removal methods, such as grinding, provided that any damage caused by the machining process is removed using electrolytic polishing.

- Results show that residual stress measurements obtained using X-Ray diffraction techniques are in good agreement with those obtained by neutron diffraction. XRD data that has been corrected for material relaxation caused by the electrolytic polishing showed improved agreement with neutron measurements.

- 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 6 and 7 for further information. Within this measurement note simplified general solutions for flat plates and hollow cylinders have been presented. These can be used for shallow depths of material removal as illustrated with all of the examples presented in this document.

- Having validated the XRD depth profiling analysis, further examples of depth profiling have been presented and excellent comparisons with hole drilling measurements have been obtained.

- Measurements on the shot peened aluminium plate showed there to be a very highly compressive layer which reached a maximum compressive value at depths of ≈100 μm. This compressive zone relaxed to approximately zero stress after a further 100 μm. In this case the agreement between the XRD and fine incremental hole drilling was excellent. If conventional hole drilling were performed then it is clear that the extra information regarding the stress profile furnished by the XRD measurements would have been lost by this approach.

- For the dry machined 321 stainless steel disc, the near surface residual stresses in the circumferential (hoop) direction were approximately five times greater than those in the radial direction. A slight offset between the XRD and hole drilling values in the hoop direction is evident, which could partly be due to zero point detection and stress distribution within the sampled area. However, agreement between both techniques is once again excellent; with high tensile stress in the hoop direction reaching a maximum of around 500 MPa just below the surface, falling to around 80 MPa within approximately 100 μm. The radial residual stresses are tensile at the surface but become compressive at a depth of ≈50 μm. A maximum compressive stress is reached at a depth of around 180 μm, which then relaxes to zero after a further 150-200 μm.

References and Further Information

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Additional Information

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