Residual stress evaluation using XRD and failure analysis

A. BAHDUR
National Metallurgical Laboratory, Jamshedpur - 831 007, India

ABSTRACT
An industrial component often contains residual stress due to processing or in-service conditions. Residual stress is important among other surface integrity parameters, because it can contribute directly to pre-mature failure of structures. The knowledge of the origin of residual stress, magnitude, type and distribution with respect to applied stresses is essential to prevent failures and to enhance the lives of the components. X-ray diffraction based technique for the evaluation of residual stress is non-destructive and determines the nature and magnitude of both macro and micro surface stress. It is based on the change in inter-planar spacings which causes a peak shift of an X-ray diffraction line. The challenge in this method lies in the determination of the X-ray elastic constants for a selected crystallographic plane. These are used to convert the measured strain data into stress values. The X-ray diffraction work has some inherent problems due to large grain size, presence of texture, strain gradient and shear stress. Ways are described to overcome these shortcomings. Special diffractometers are mentioned which are portable, fast and use position sensitive detectors. They can be utilized for carrying out in-situ work at plant sites. The latest research for measuring sub-surface stress, stress gradients, stress at elevated temperatures and in different environments are briefly outlined. Finally, some case studies dealing with the failure analyses of components are highlighted.

INTRODUCTION
Failure can lead to disastrous consequences. Their prevention is important. Evaluation of residual stress (RS) can locate precursors to component failures, thus preempt and avoid catastrophic failures. RS being a sensitive indicator for variations of processing parameter and component properties, serves as a practical method for life extention.

A free body contains residual or locked-in stress due to processing or in-service
conditions. RS occupy a key position among other surface integrity parameters because they can contribute directly to premature failure of metallic and ceramic structures. Therefore, knowledge of their origin, magnitude, type and distribution with respect to the load induced stresses is of great importance and a pre-requisite if changes in the stresses are intended. Generally, residual tensile stresses at the surface are detrimental and may lead to fatigue and other structural failures, when the service stresses are superimposed on the already present RS. The presence of RS is generally not recognized. The methodologies of measurement, the costs and difficulties encountered by the usually distracture techniques have sometimes discouraged their intensive measurements for a better utilization. Nonetheless, ignoring the problem can be dangerous in an increasing number of situations. These encompass the need of mass reduction in structures, reduction of load safety margins, more stringent quality specifications and the application of fracture mechanical calculations.

The devastating failures of liberty ships in the US in the early forties was due to RS and a microstructure that did not impart ductility to the material. Also, I-beams split violently due to a change is RS from compressive to tensile by skew cuts, leading to crack initiation and propagation. The failures related to RS may not be as spectacular now but they do cause economic burdens on industries.

Fatigue and stress corrosion cracking are the two major causes of failures in industrial components due to the presence of tensile RS [11]. Hydrogen embrittlement is also strongly affected by the presence of tensile macro and micro stresses. Compressive stress, though harmless, generally poses problems, since for equilibrium, the compressive RS in one locations must be balanced by a tensile RS in another. Thus, on removal of the RS (e.g. by grinding, pitting etc.), tensile RS may appear on the surface leading to failures. Besides, components buckling due to compressive stresses is a common phenomenon [12].

MEASUREMENT OF RESIDUAL STRESS

Experimental determination of RS in a component is an important tool for process control, quality control, design assessment and failure analysis.

Several experimental techniques have been evolved for the measurement of RS based on diffraction, mechanical, optical, magnetic, ultrasonic and others [3]. The mechanical techniques are destructive and depend upon the removal of material from a component and monitoring the changes in strain distribution due to stress relaxation. The most useful and versatile technique is based on the change in spacings between parallel set of crystallographic planes due to macro stress that causes a shift in the peak of an X-ray diffraction line. Micro stress on the other hand, changes the width of the diffraction line.
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(a) Vector diagram of plane spacings $d$ for a tensile stress $\sigma_o$

(b) Measurement of $d_n$

(c) Measurement of $d_i$

Fig. 1: (a) Plane-spacing diagram. (b) and (c) Orientations of X-ray beams relative to specimen. $N_s$ = normal to specimen surface, $N_p$ = normal to reflecting planes.

Fig. 2: Stresses at the surface of a stressed body. The stress to be measured is $\sigma_\phi$. 

[Diagram of vector diagram, plane-spacing diagram, and orientation of X-ray beams]
XRD Technique

X-ray diffraction (XRD) based technique is non-destructive. Repeated measurements are therefore possible at different stages in service life of a component. It determines the nature and the magnitude of both macro and micro surfaces stress (~20 mm in steel). The majority of failures of industrial components initiate from the surface where the stress and the ambient conditions are more stringent than at the interior. This makes measurements by XRD relevant, practical and applicable for industrial problem solving. Other advantages of XRD technique are that stress-free sample is generally not required. It is possible to measure absolute value of stress in individual phases, localised stresses and steep stress gradients.

XRD is the only technique to give stress in individual phases. It provides information on the metallurgical conditions of the sample e.g. grain size, preferred orientation, surface cold work or hardness and process changes. It is very fast, precise and gives reproducible results. The accuracy of stress value depends upon the accuracy in the determination of 2θ i.e., shift in the diffraction peak position due to stress, by orienting the sample at different ψ angles w.r.t. the incident X-ray beam, Fig. 1. Thus a series of measurements can be made with the planes of the atoms oriented at different angles w.r.t. the sample surface, and hence the surface stress, Fig. 2, this is known as multiple exposure (Sin²ψ) method. A straight line relationship is generally observed in the plot between peak shift and Sin²ψ and the slope of the line is given by

\[ m = \frac{(1 + \nu)}{E} \cdot \sigma_\Phi \]

where E, ν are Young's modules and Poisson's value for the particular crystallographic plane used. Consequently, stress in any chosen direction on the surface of the specimen is

\[ \sigma_\Phi = \frac{E}{(1 + \nu)} \cdot \cot \theta \cdot \frac{\Delta \theta}{\sin^2 \psi} \cdot \frac{\Pi}{180} \]

Fortunately, angle φ does not occur in the above equation. The direction of the surface stress can be made to coincide with the longitudinal, transverse circumferential or any other arbitrary direction. The magnitude and direction of principal stress can be determined by making four measurements at do, di at φ and φ ±60°.

Determination of X-Ray Elastic Constants

The X-ray method looks only at a particular crystallographic plane. The mechanical elastic constants are averaged over all the orientations and can not be used
in x-ray work. A vigorous theoretical calculation of X-ray elastic constants (XEC) requires a complete theoretical solution of the influence of elastic anisotropy and grain interactions on X-ray strain measurements. However, the theoretical XEC values may not agree with experimental ones due to the dependence of XEC on composition and second phase components [5] ii) grain size [6] iii) microstructure iv) deformation v) heat treatment. The magnitude of these effects depends on the hkl reflection used.

The experimental determination of XEC can be done on a rectangular beam specimen with strain gauges and generating the calibration curves on dead weigh loading in a 4-point bend fixture, Fig. 3 [7]. Knowing the applied load, sample dimensions and moment arm of the 4-point fixture, the actual stress in the outer fibre of the sample is calculated. On the diffractometer, the fixture is clamped to minimise displacement errors. The lattice spacings of the selected set of planes is measured five or six times at ψ angles of 0 and 45°, at the same stress levels (as used in calibration) by loading. The peak shift was calculated between ψ = 0, 45° for each measurement and plotted against the applied stress. The slope of the line was determined by the least square. The elastic constant is

\[
\frac{E}{(1 + \nu)} = m \sin^2 \psi do
\]

Checks on stress data

Large oscillations in \( \sin^2 \psi \) vs. \( d \) curve occur due to the presence of inhomogeneous stress/strain data within the material. As different grains are inspected at each tilt, erroneous results can be obtained with two tilt procedure. To minimise the
The effect of gradients, high \( \psi \) points are used in the analysis. Poor precision is obtained unless the grain size in the material is right. The effect of large grain size can be overcome by sample oscillation to ensure exposure of a larger areas, and thus, averaging over many grains. In a textured or plastically deformed material, use of high multiplicity planes may have a higher probability of being oriented in a random manner and give better results. Linear \( d \) vs \( \sin^2 \psi \) data have been obtained in a textured steel using 732+651 peaks of ferrite with Mo radiation [8].

The presence of splitting in positive and negative \( \psi \) profiles in \( d \) vs. \( \sin^2 \psi \) curve indicates, that in addition to the strains in the plane of the surface, the shear strains are finite within the irradiated volume.

In order to obtain an unambiguous results from the X-ray measurements, experience and good understanding of the experimental and material conditions are required.

**Special Diffractometers**

The diffractometer technique has replaced the earlier film technique. The lab equipments consist of a fixed X-ray tube and movable sample stage and detector. With horizontal type of goniometer, much heavier and bulkier samples can be investigated. However, such diffractometer have a limited capacity for determining stress on actual industrial components.

Portable X-ray stress analysers are available with miniature X-ray tube. An effective way of reducing the data gathering and analyzing for stress measurements is the use of position sensitive detectors (PSD). They examine X-rays diffracted over \( 2\theta = 14^\circ \) simultaneously. PSD reduces the cost drastically due to shorter measuring times. The specimen is self supported and the goniometer is rigidly positioned w.r.t., the measuring point of the specimen. Since the counter need not be rotated relative to the X-ray tube during peak determination, light weight, compact diffractometers of which an electronics module is an integral part are now available. The reliable determination of peak shift between two measured intensity curves is done efficiently and accurately by cross-correlation function. As this method is a statistical one, which determines the relative peak positions, the results are undisturbed by peak splitting, irregular background intensity variations, scatter and even by slight adjusting errors of the goniometer. Since the advent of portable XRD equipment, this technique can be used on even larger or irregular shaped components in many different environments. It is being used for process control, quality control and failure analysis. Special hardware facilities have been added to the diffractometer e.g., a unit of displacement and rotation for the specimens. It is used
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for automatic surface scanning with local stress measurement in different specimen orientations. In connection with the slope etching method, the same unit is also used for the determination of RS profiles below the surface, making an automatic stress depth analysis possible. The technique has kept pace with the needs of the researchers in the area of multiphase materials and composites. The new developments enable stress gradients and in-situ stress at elevated temperatures and in different environments to be determined \(^9\).

CASE STUDIES ON FAILURE ANALYSIS

In-situ measurements of RS on a component while in operation is very important but not always possible. In some cases, where failure of components has already taken place, the failed component can be brought to the lab and RS can be measured on it. The stress profile may have altered near the failed region, nevertheless, the measured stress values give a fair indication of the nature and level of stresses existing prior to the failure. This helps in evaluating the cause of failure and recommendations can be made to improve the lives of the remaining similar components. Some case studies would be discussed during the presentation.

REFERENCES