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TITLE: RESIDUAL STRESS MEASUREMENT USING THE PULSED NEUTRON SOURCE AT LANSCE

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Residual stress measurement using the pulsed neutron source at LANSCE

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Abstract

The presence of residual stress in engineering components can effect their mechanical properties and structural integrity. Neutron diffraction is the only measuring technique which can make spatially resolved non-destructive strain measurements in the interior of components. By recording the change in the crystalline interplanar spacings, elastic strains can be measured for individual lattice reflections. Using a pulsed neutron source, all the lattice reflections are recorded in each measurement which allows anisotropic effects to be studied. Measurements made at the Manuel Lujan Jr Neutron Scattering Centre (LANSCE) demonstrate the potential for stress measurements on a pulsed source and indicate the advantages and disadvantages over measurements made on a reactor.

Introduction

Residual stress measurements by neutron diffraction with spatial resolutions of a few millimeters in engineering components were first made in the early eighties on reactor sources [1,2]. However as the flux and reliability is improved at pulsed sources like LANSCE and IPNS in the United States and ISIS in the United Kingdom, their original limitations are diminishing and it is becoming feasible to define and examine small sampling volumes in components. The demand for beam time at reactors remains high so pulsed sources can provide a useful and in some cases superior alternative.

In a standard diffraction experiment all the lattice reflections of a powder specimen are usually required. Typically reactors provide a continuous source of thermal neutrons which can be monochromated to provide relatively intense beams of collimated neutrons. By moving a detector around a specimen an intensity (2θ) scan can be obtained at a monochromatic wavelength. Monochromation of a neutron beam from a reactor uses only a small fraction of the available neutrons to produce the diffracted spectrum, however the intensity of even a monochromated beam is considerable.

By contrast at a pulsed source the integrated neutron flux over any wavelength range is much lower than on a reactor. However it is inherent in the operation of a pulsed source that all of the neutrons in each pulse can contribute to the measured spectrum making efficient use of the available neutrons. Each pulse represents a wavelength scan which is analogous to scanning in 2θ from 0 to 180°. This efficiency and because the whole of the diffraction pattern is required makes the timescale for measurement on pulsed sources comparable with reactor sources.

Efficiency of measurement is crucial for examining components which involve small sampling volumes and large path lengths. The feature that works against a pulsed source in achieving comparable count times with steady state sources is that macroscopic strain measurements can often be inferred from just one or two lattice reflections. Thus only a small 2θ range is scanned and the pulsed source count times are long in comparison to reactors. Nevertheless measurements at LANSCE made in 1990 indicate that the intensity of pulsed sources

has reached a level where it is feasible to define small (<100mm^3) sampling volumes in plate-like specimens and to make measurements in hours rather than days. A number of features have made this possible including the summation over all of the angle subtended by the 90° detectors, measurement in more than one scattering geometry, and analysisi by the use of profile refinement.

The principle of measurement on a pulsed neutron source is identical to a steady-state source but practical differences and difficulties exist because of the time of flight analysis of the diffracted neutrons. Apart from the experimental difficulties pulsed source measurements conveniently provide a comprehensive sample of the strain for all the measurable lattice directions. This information can elucidate both the grain interaction stresses and help to validate the use and selection of single reflections at reactor sources. The data can also be used to test the different models of material deformation.

Measurements at LANSCE demonstrated that a sampling volume of less than 40mm^3 could be defined in a flat specimen while maintaining sufficient neutron intensity to make measurements even after attenuation through 20mm of steel. Changes in lattice spacing in steel of 50 μstrain were resolved giving a stress discrimination of ~10MPa. The work described in this paper was performed using the Neutron Powder Diffractometer (NPD) at LANSCE although preliminary measurements on a 5mm thick steel ring were attempted at ISIS [3,4].

Anisotropic strain effects

Even in the elastic regime the manner in which solid polycrystalline materials deform is not well understood. Measurements using a stress rig have shown that the strain state in a single phase steel is far from the homogeneous assumption invariably used in engineering design [5]. When intergranular effects, elastic anisotropy and the anisotropic yield in grains of different orientations are considered, it is clear that the theoretical situation is very complicated

Theoretical limits were proposed by Reuss[6] and Voigt[7]. Reuss proposed that each crystallite experienced the same stress field while Voigt suggested that each crystallite experienced the same strain field. The Voigt model implies that no strain anisotropy will be observed for different lattice reflections while the Reuss model predicts that strains in the polycrystalline aggregate will depend on the orientation of the crystallites with respect to the applied stress. A more realistic model was proposed by Kroner [8] in which crystallites are assumed to be embedded in a homogeneous elastic medium with elastic properties equal to those of the bulk material,. The Kroner model has been applied to measurements made at LANSCE and is described elsewhere in this volume [9]. The implications of the three models are discussed by Sayers[10]. The other model which is receiving considerable attention particularly in the context of composite materials is that of Eshelby [11]. Checks and validation of the different models require the measurement of the strain response of many different lattice reflections.

Experimentally measurements are complicated by the presence of interstitial impurities such as nitrogen or carbon which affect material properties, as well as uncertainties resulting from voids, relaxation, phase transformation incompatabilities and changes in cohesion at grain boundaries. On relief of an applied load, grains in different orientations which had different yield stresses can be driven into compression or held in tension. These effects constitute microstresses and exist over length scales equivalent to the grain size in the material.

Diffraction measurements of residual lattice strains examine the elastic strain present in the grains contributing to the diffracted maxima. However this may result both from the macroscopic strain effects or from grain interaction effects which may be loosely associated with type 1 and type 2 residual stresses[12]. It is important to make the distinction since it is usually the type 1 stresses which concern the engineer. The benefits of a pulsed source in the measurement of grain interaction stresses [13] were demonstrated in 1983 but hitherto we are unaware of any spatially resolved strain measurements in solid components at pulsed neutron sources.

On a reactor source when one lattice reflection is examined only a small number of grains are examined with the risk that the selection may not be representative of the bulk material or may represent a special case. Although in many cases the use of a single reflection to infer macroscopic (type 1) residual stresses has been justified, if there is any reason to suppose that grain interaction

stresses may be significant then there is a case for making a more detailed survey of the lattice reflections which may beneficially be performed at a pulsed source.

Operation of a pulsed source

At a pulsed source neutrons are produced by spallation, which occurs when energetic particles interact with target nuclei. Heavy element targets offer the best efficiency for emitted neutrons per unit energy of the incident pulse. At LANSCE proton bunches are accelerated to 800MeV then are directed at a tungsten target. The target nuclei are excited and "boil off" neutrons and fragments of the target nuclei in an evaporation process. Each incident proton bunch produces a pulse of highly energetic neutrons. For diffraction experiments the fast neutrons from the initial pulse must be thermalised using a moderator.

The difference between a pulsed and steady state source concerns the manner in which the scattered radiation is detected. If a neutron is created at a known time and position (i.e. when the proton pulse interacts with the target) its wavelength on detection can be determined from the distance and time it took to travel to the detector. Thus no collimation between the detector and specimen is needed because the wavelength is inferred from the geometry of the scattering process and the time of flight (TOF). Each pulse contains a continuous spectrum of energies thus the Bragg equation for all the lattice planes will always be satisfied by all directions. By correcting for slightly different scattering geometries a final spectrum can be constituted from the summation of the many individual spectra from individual pulses and from individual detector tubes. Thus lattice spacings, d_{hkl} are determined by maintaining a constant diffraction angle and scanning the wavelength. The wavelength is inversely proportional to the velocity thus the TOF, t, is proportional to the wavelength, λ, and the lattice strain is given by:

$$\frac{\Delta d_{hkl}}{d_{hkl}} = \frac{\Delta \lambda_{hkl}}{\lambda_{hkl}} = \frac{\Delta t_{hkl}}{t_{hkl}}$$

The diffracted maximum intensity for each lattice spacing occurs at a discrete wavelength. The strain is determined from the change in TOF between the measured value and unstrained material.

The easiest way of improving the resolution of a spectometer on a pulsed source is to increase the path length or the time of flight between the target and the detector. However the improved resolution is often compromised by beam losses along the flight path and ultimately for long guides "frame overlap" occurs when the slow neutrons from one pulse are overtaken by fast neutrons of its successor. Since the measurement of engineering strains in components is usually limited by the intensity of the neutron beam the use of the highest resolution spectrometers at pulsed sources are probably not warranted. On the other hand medium resolution spectrometers like the NPD at LANSCE or POLARIS at ISIS have good intensity while still having adequate resolution

Definition of a sampling volume

A detailed description of the manipulation and collimation system (MACS) employed at LANSCE is included elsewhere in this volume. However a brief discussion of the collimation is included here.

One of the advantages of the NPD is that it has 4 detector banks at ±90° and ±148° which permit 4 simultaneous strain measurements. The NPD is 32m from the target and the divergence of the incident beam at the sample position is small. Consequently the distance between the incident aperture and the specimen is not crucial. The penumbra produced by a square incident aperture with edges 2mm placed 100mm from the specimen is ~0.2mm in each dimension.

By contrast the position of the exit aperture is critical to the dimension of the sampling volume along the incident beam because there is no inherent collimation between the sample and the detectors. In principle soller collimation could be installed but it is essential to maintain a large

solid angle of detector visible at the specimen if the count times are to be kept reasonable. The requirement that most or all of the NPD 90° bank is accessible to the sampled volume combined with the requirement of millimeter spatial resolution along the incident beam constrain the exit collimation to be placed as close to the incident beam direction as possible (typically less than 30mm).

The general situation for an aperture of width d, thickness t and distance L from the incident beam path (ignoring the finite size of the incident beam) is shown in figure 1. The drawing is not to scale and typically L will be 20mm, d ~3mm and t~13mm. In practice the angular spread subtended by the 90° detectors on the NPD is ~11°.

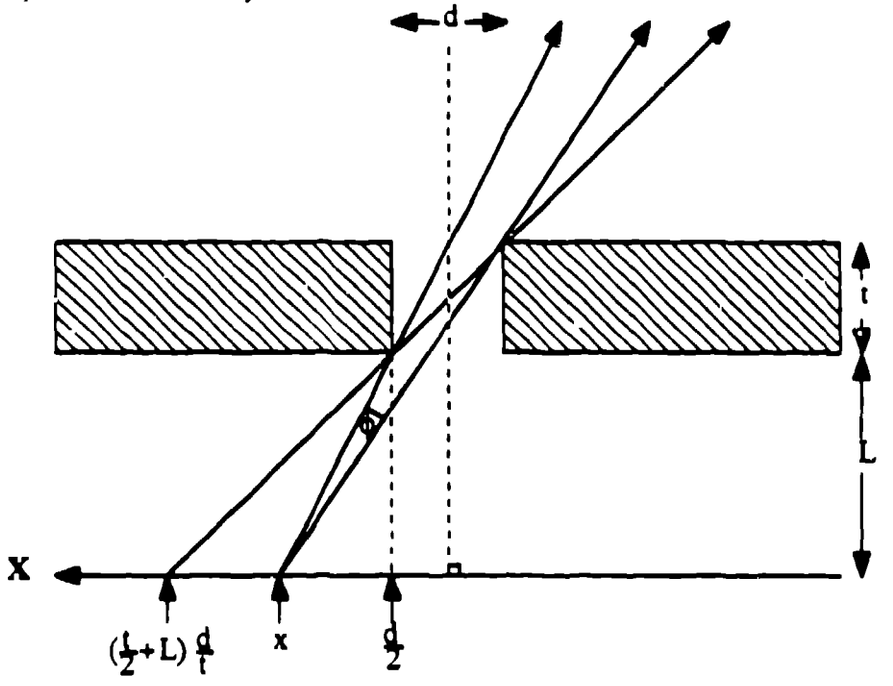

Figure 1: Resolution along the incident beam

The aperture width, d, and to some extent its thickness, t, are not easily changed however the distance, L should always be minimized. The spatial resolution along the incident beam decreases with increasing distance of the exit aperture from the incident beam.

For the common case of plate specimens inclined at 45° to the incident beam direction masks have been fabricated with the aperture at 45° to their surface. These masks are supported from rails can be moved adjacent to the specimen surface which minimizes the dimension L with a commensurate improvement in figure 2.

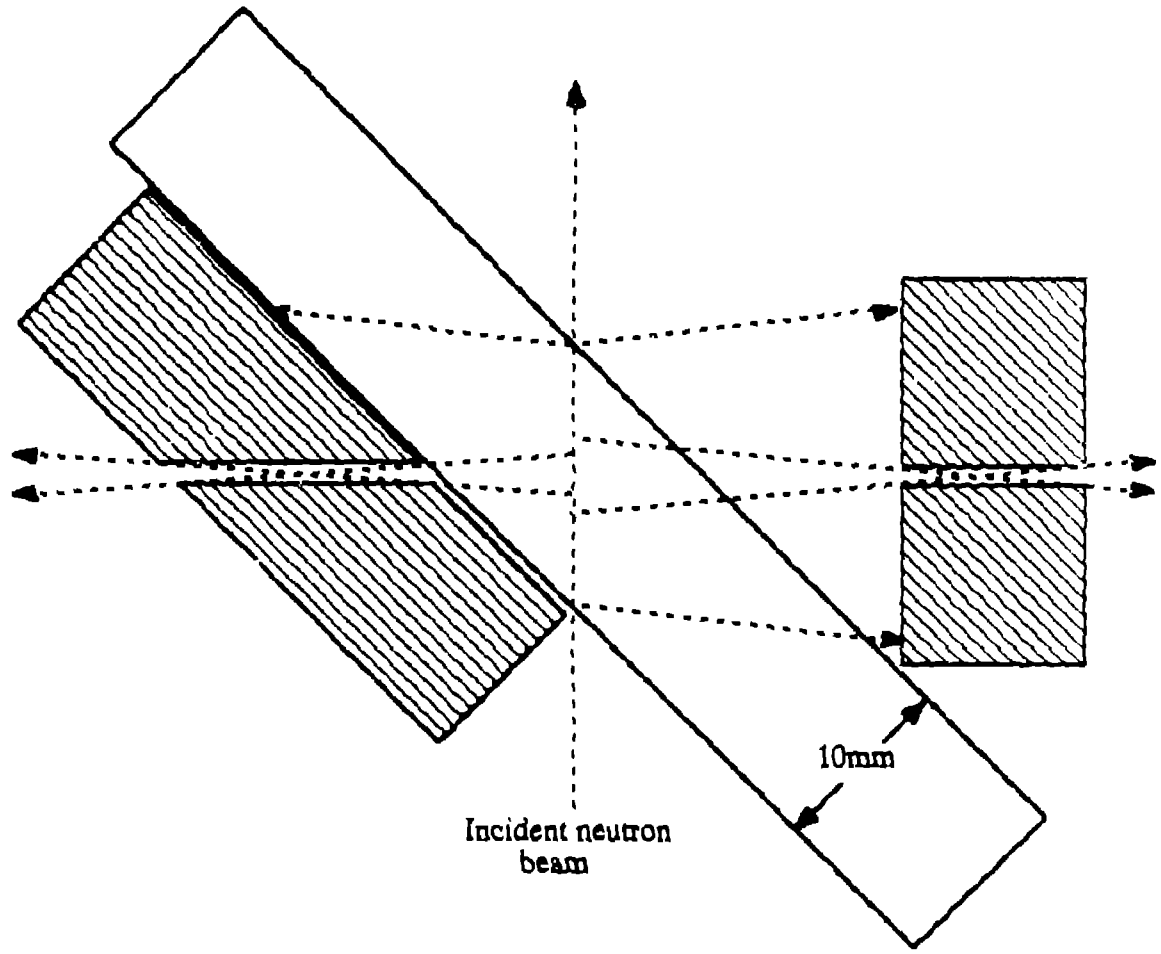

Figure 2 Improvement in resolution for 45° apertures

The two 90° bank consist of 16 helium tubes which subtend ~11° at the sample position. The spectra obtained in individual tubes correspond to slightly different strain directions but accumulate too slowly to give adequate statistics. Consequently the spectra from all of the detectors are summed to give an integrated spectrum corresponding to a measurement with a spread in scattering vector (and thus of the strain measuring directions) of ~5.5° (fig 3). This average is not large when considering macroscopic engineering strains but is necessary to obtain favourable count times.

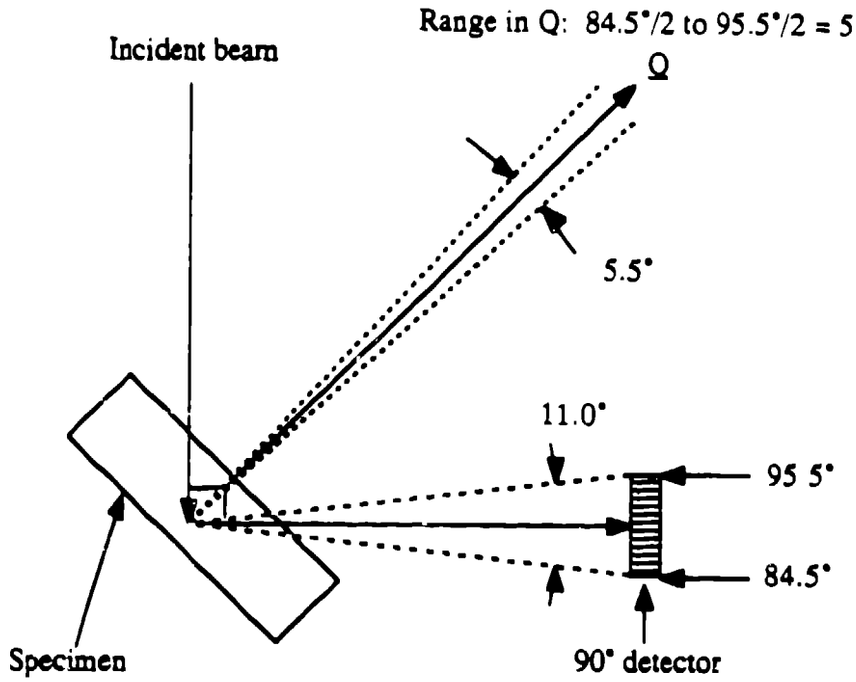

Figure 3 Average in Q by summing over 90° detector

Calibration

One unique problem to the pulsed sources concerns the calibration of the detectors for a particular sampling volume. On a time of flight instrument the sampling volume can be defined at any position within or along the incident beam but it is necessary to calibrate the detectors for scattering from that point. In general a well characterised calcium fluoride powder is used for which the lattice parameters are well known and thus the path lengths can be predicted from the sampling volume to each detector bank .

The sampling volume is defined by the intersection of the incident and diffracted neutron beams. If an aperture is moved in a direction which is not parallel to the incident or 90° diffracted beams the sampling volume will be displaced in space. Not only does this introduce an ambiguity into the position of the sampling volume in the specimen but because the detectors are calibrated for a sampling volume centered on one specific point a constant error is introduced into the time of flight spectrum. On the NPD errors associated with ~1mm displacements of the sampling volume are equivalent to constant strain errors of ~30μstrain. If the sampling volume is unknowingly displaced by more than a few millimeters, then a significant strain error can be introduced. Since the apertures are frequently moved or replaced over the course of an experiment, care must be taken if repeat calibrations which would take up valuable beam time are to be avoided.

For the 1991 run cycle at LANSCE an optical alignment system will be installed for the MACS equipment. It will consist of two alignment telescopes mounted on a dummy sample chamber which is identical to the real sample chamber on the instrument which has no optical access. The intersection of the directions defined by the telescopes mounted at 90° to one another will identify the centre of the sampling volume and will ensure that the apertures have not been displaced. For correct placement of the apertures the telescope directions will be placed accurately parallel and normal to the beam direction. When the system is implemented it is expected that the specimen and aperture positions will be reproducible to within 0.1mm.

Data analysis by profile refinement

Bragg reflections in each spectrum can be fitted individually to give strains for grains in different orientations relative to the direction of the scattering vector enabling the examination of anisotropic effects. This is analogous to individual measurements using a steady state sources except that no reorientation of the component is necessary to obtain data for all of the lattice reflections in the same direction in the component. At LANSCE a program is available which fits, calculates and tabulates the strains of all the reflections in each spectrum (after the initial positions have been identified).

An alternative for analyzing the data is profile refinement. If the crystal structure is known then the intensities and positions of the observed lattice reflections can be predicted using the Rietveld method of profile analysis[14]. By making a least squares fit between the observed and predicted profiles, the atomic positions and lattice parameters for the material can be determined. In regions of compression or tension the lattice parameter will change and can be used to infer the strain.

The use of profile refinement is advantageous for several reasons. For cubic materials (which have one lattice parameter) the accuracy with which the lattice parameter is determined in a profile refinement is better than the accuracy for the fit of an individual reflection because all of the measured reflections contribute to the refinement. This is true despite the anisotropic elastic strain effects that occur for different reflections. Thus the profile refinement does not take into account the difference in compliance exhibited by different directions in a crystal lattice. It ignores any deviation from perfect crystalline behaviour and fits the best possible model to the data. In the absence of preferred orientation, the lattice parameter can (arguably) be assumed to offer the best estimate of an isotropic strain for engineering calculations.

A second advantage of profile refinement relates to the issue of count times. Since refinements use the data from all the lattice reflections, reasonable refinements are obtained long before the accuracies of fits of individual reflections approach usable values. If the anisotropic strain information for individual reflections is not required then count times can be substantially reduced permitting measurements from small sampling volumes to be made in times approaching those on steady state sources. When the count times are long enough to give reasonable accuracies for the fits of individual peaks the accuracy specified for the lattice parameter is often better than +-25μstrain. One future possibility would be to modify the Rietveld refinement to accomodate the elastic anisotropy but at this time the analysis is better served by ignoring it.

Examples

Deformed Austenitic Ring

Calibration measurements were made on a plastically deformed ring to assess the count rates and feasibility of defining a small sampling volume using the NPD. The specimen was previously examined at a steady-state source as part of a program to validate a finite element calculation[15]. A detailed description of the measurements is given elsewhere where they are compared with values predicted by the Kroner model[9]. The experimental parameters are described here to illustrate the count times that are possible for flat specimens and to show the agreement obtained between the strains obtained from the lattice parameter and from the fits of individual reflections.

The ring dimensions were 76mm internal diameter, 127mm outer diameter and 13mm thick. It was plastically deformed by diametral loading in compression which introduced a residual stress pattern similar to a bent beam. Measurements were made along a radius in a section at 90° to the previously applied stress direction. The ring was placed at 45° to the incident beam so that normal strain directions were measured in the plane of the ring and normal to it. Depending on the orientation of the ring either the hoop and axial or the radial and axial strains were measured simultaneously in opposing 90° banks. Using a sampling volume of 40 mm^3 at a beam current of ~75μA two strains were measured every 4 hours. Additional information was recorded on the back scattering detectors although no collimation was used. A spectrum from one of the 90° banks is shown in figure 4..

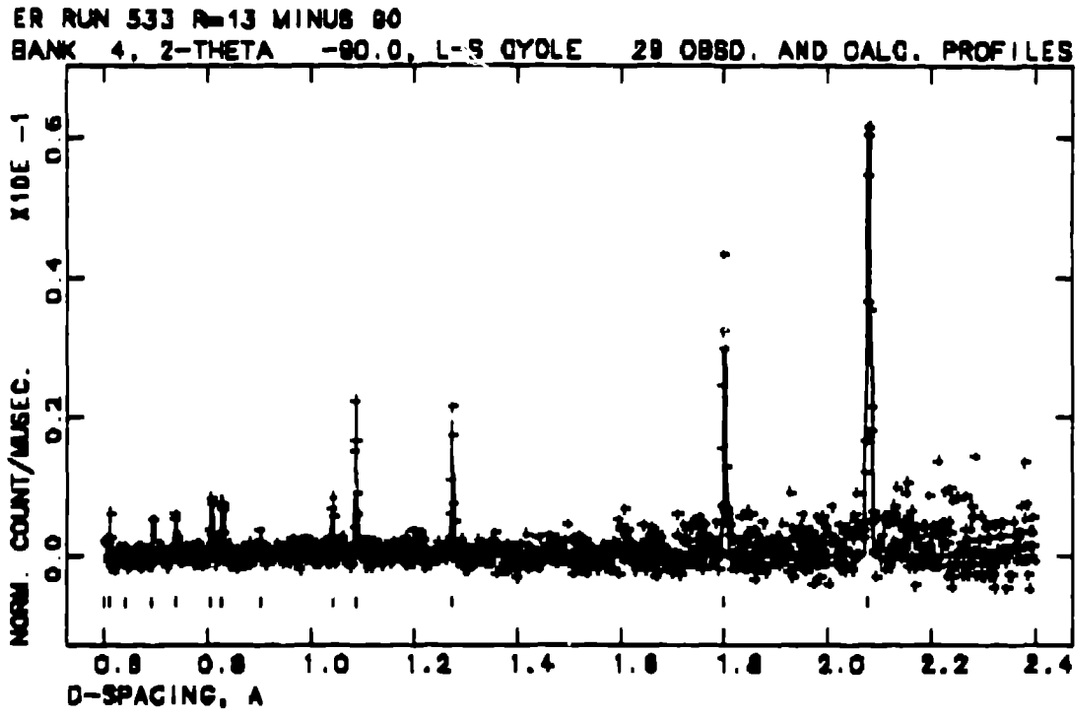

Figure 4 NPD 90° spectrum obtained in 4 hours 32mm^3 path length through austeintic steel ~18mm.

With count times of 4 hours the accuracy for the peak fits of strong individual reflections was ~±80μstrain. This compares to an accuracy specified for the lattice parameter from the profile refinement of ~+- 30μstrain. In figure 5 the tangential (hoop) strains are plotted for 5 reflections of differing elastic compliance together with the strains predicted by the profile refinement. The strains by lattice parameter are connected by a solid line. The solid line is bounded by the stiff (220,111) and compliant (200) directions which reflects the averaging effect of the profile refinement on the elastic anisotropic effects. The smooth variation of the tangential strain predicted by the lattice parameter gives credence to its validity as a bulk average and to the comparatively small errors predicted by the refinement.

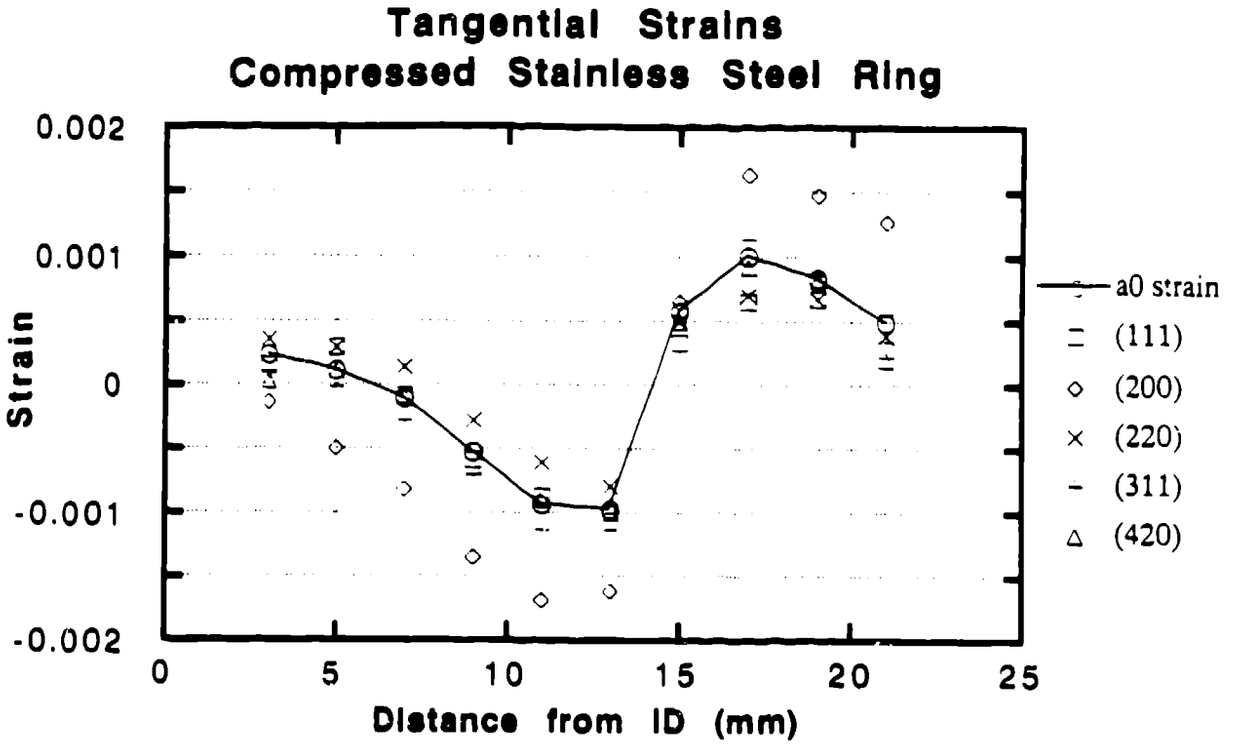

Figure 5 Hoop strains by individual reflection and by lattice parameter

The strains shown above were obtained from the combined spectrum at each position obtained by summing all 16 individual detectors in each 90° bank. To assess whether profile refinement can be used to infer the strain in shorter periods we summed progressively fewer detectors and examined the errors cited for the lattice parameters from repeated profile refinements. The results are shown in figure 6. By summing only 4 of the available 16 detectors a 1 hour counting period was simulated. The error at 1 hour for the lattice parameter of = +- 50 μ strain is reasonable. Of course the peak fits on individual lattice reflections are poorer and the error for the strongest reflection was =+- 120μstrain. Nevertheless if the lattice parameter is sufficient then for specimens where advantage can be taken of the opposing 90°banks count times for a 32mm^3 sampling volume can approach 30 minutes per strain direction.

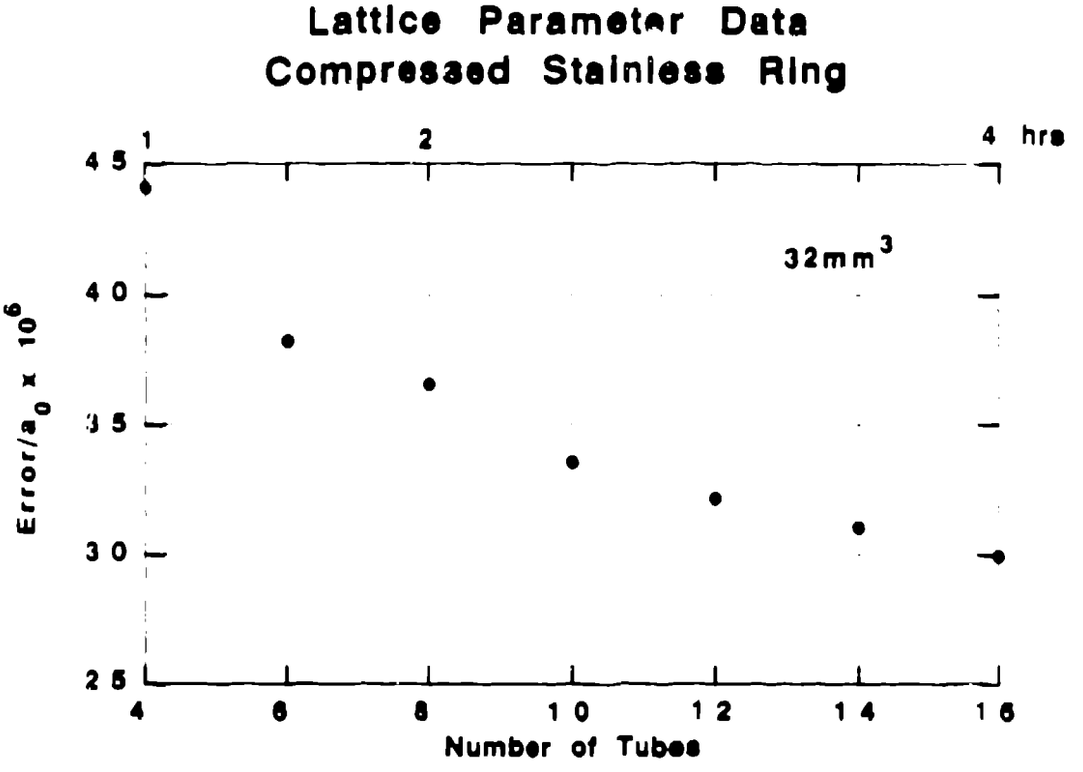

Figure 6 Error in lattice parameter vs # of detector tubes i.e. count time

Other examples

Another experiment, which took advantage of the characteristics of a pulsed source, addressed the effectiveness of vibratory stress relief in reducing the residual stresses near welds. Two multipass welds were examined in 300mm by 300mm by 25mm thick austenitic steel plates one of which was subsequently stress relieved. Strong texture changes occurred as the sampling volume was moved through distances as small as 4mm through the weld and heat affected zones because of epitaxial growth of crystals from the melt and because of the differences produced in sequential weld beads. However because all the lattice reflections were examined in each spectrum the irregular absence of some reflections was not a major problem.

By taking advantage of the symmetry along the welding direction an elongated "matchstick" sampling volume (70*4*4 mm) was used with usable spectra being obtained in 3-4 hours. The preferred orientation was too severe to make refinements of the data so the strain information was deduced solely from the fits of individual reflections. The errors obtained for individual peak fits were typically ~+-100μstrain. However by calculating the lattice parameter from a least squares fits just of the peak positions (ignoring the intensities) it is expected that more accurate strain information can be obtained from the data.

Other preliminary studies addressed whisker and particulate reinforced Al-SiC composites , compacted powders and an autofrettaged ring. In the experiments on the metal-matrix composites uniaxial tension and compression specimens were deformed to differing levels of plastic deformation. On release of the applied loading the residual strain state of the two components in the composite were examined. These experiments did not require definition of a small sampling volume because the grain interaction strains of interest were assumed to be uniform throughout the samples. Neutron diffraction is valuable for studies of this nature because the matrix and reinforcement can be examined simultaneously and non destructively. In addition relatively large specimens can be examined making count times comparatively fast.

Discussion

The advantages of the time of flight method are: that all the lattice reflections are recorded with the same resolution in each spectrum, that simultaneous strain measurements in different directions are possible, that a spectrum can be recorded at any angle (subject to the geometry of the component and that multiphase materials can easily be examined. The disadvantages compared to monochromatic measurements are; that good volume resolution is only possible by placing the diffracted beam apertures close to the specimen, that calibration is harder and more time consuming and that count times are generally longer.

Studies in materials exhibiting strong texture variations or in multi-phase materials are particularly suitable for a pulsed source because all the lattice reflections are collected in each detector bank. Examples include the study of grain interaction strains in metal matrix composites and the unexpected appearance of a weak ferritic second phase in the stainless steel weld material. Using monochromatic neutrons to follow texture changes or to examine extra phases necessitates changes in wavelength and orientation to keep the scattering vector correctly aligned relative to the specimen.

Sampling volumes in the range 30-50mm^3 were routinely defined on the NPD and [illegible] case as small as 8mm^3 (in a 5mm thick ring). However in all cases the specimens had flat parallel surfaces against which the exit apertures could be placed. If the principal axes are not assumed specification of the stress tensor requires a minimum of 6 independent strain measurements. This will require the rotation of specimens and may compromise the sampling volume if the apertures must not be moved. The only solution to this problem is to use radial soller collimation which would permit the definition of a sampling volume at a distance but this remains unproven technology.

The uncertainty concerning the count times to give specified strain accuracies for different materials, specimen geometries and aperture geometries reflects the paucity of experience in defining small (< 100mm^3) sampling volumes on the NPD. However if strains can be inferred from the lattice parameters of Rietveld refinements and if simultaneous strain measurements can be made in different detectors the count times are not dissimilar to measurements on steady state sources. Of course the option of simultaneous strain measurements in different detectors is dependent on the specimen geometry and may not always be possible.

Determination of the unstrained material response remains a problem both in general and because changes in path length cause shifts in the TOF spectrum which can be misinterpreted as strain variations. Its measurement on a pulsed source in small off-cuts or annealed specimens has all the same uncertainties which apply to a steady state source. One extra option for estimating the unstrained value is possible on a pulsed source if the assumption is made that there is no hydrostatic stress in a region where the stress is changes from tension to compression. In the deformed ring described above an elastic core exists approximately between 11 and 17mm from the bore of the ring. In this region the stress in the hoop direction varies from compression to tension. Strains can be calculated using an arbitrary unstrained value and then the data for different reflections can be plotted against the stiffness anisotropy factor, A_{hkl}^2 where

$$A_{hkl} = \frac{(h^2k^2 + k^2l^2 + l^2h^2)}{(h^2 + k^2 + l^2)}$$

The strains differ due to the anisotropy of the elastic compliance in different directions within the unit cell. When the strains for different reflections in one measuring position are plotted against A_{hkl} the slope is approximately linear but changes in magnitude with the stress and in sign between the tensile region and the compressive region. An example is shown in figure 7.

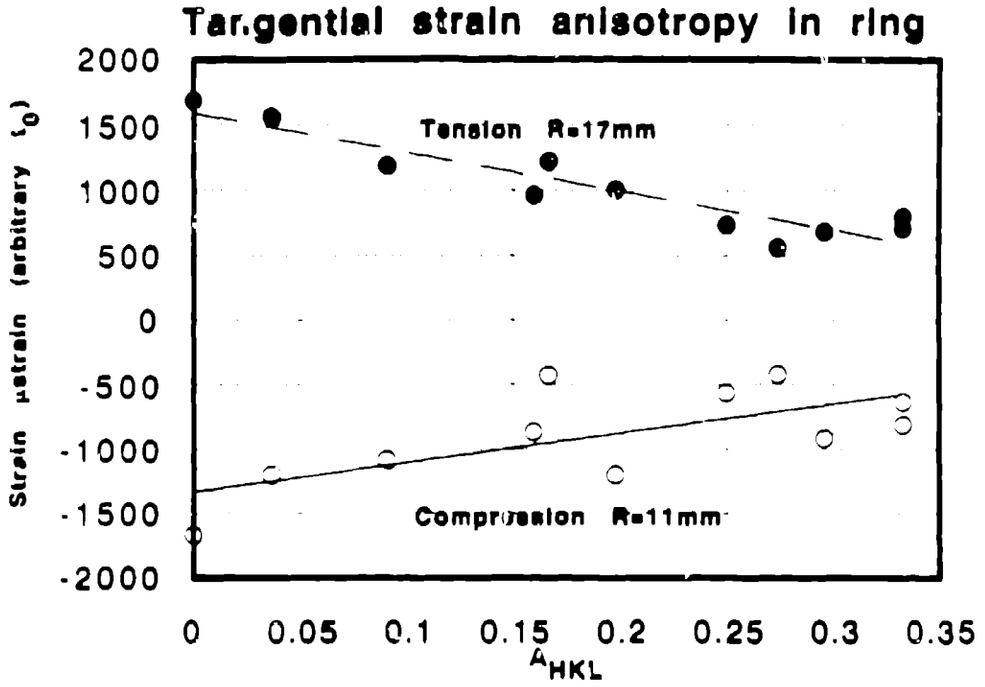

Figure 7 Strain anisotropy at two positions in compression and tension

The details of the gradient of the slope are complicated but if the change in sign is assumed to indicate the change from compression to tension then if a measuring point is identified in the ring where the gradient is found to be zero then it is reasonable to assume that the material at that point is strain free. The approach is limited in its application but may occasionally prove useful and is easy to apply for pulsed neutron data where all the reflections are available.

Much calibration work remains to be performed using the new MACS set-up on the NPD. In particular the sensitivity of the CaF_2 calibrations to the size and shape of the sampling volume. The definition of the sampling volume using boron nitride apertures is predictable by geometric considerations however the effects of strongly absorbing materials in weighting the result towards the incident side of the sampling volume need to be considered. If radial soller collimation can be engineered then the biggest limitation of the measurements on a pulsed source which requires the proximity of the diffracted beam apertures to the incident beam path could be circumvented. This needs consideration.

Conclusions

Incident and exit beam collimation have been installed on the NPD at LANSCE permitting spatially resolved measurements to be made in solid components. Good spatial resolution can only be achieved in plate specimens but where this is the case the NPD provides strain information from all the lattice reflections in count times which are not dissimilar to a steady state source. Situations where grain interaction strains are being investigated in multiphase materials are particularly appropriate for spectrometers at pulsed neutron sources. All three of the main pulsed sources, LANSCE, ISIS and IPNS have spectrometers with adequate resolution and flux for residual strain measurements.

On the NPD diffracted beam collimation has only been installed on the ±90° detectors but strains are also recorded on the back scattering detectors providing extra information. For plate

specimens using the two opposing 90° banks two strain measurements (normal to each other) with defined sampling volumes are obtained in each measurement. The use of profile refinement can reduce the count time required to obtain acceptable strain accuracies.

The relationship between macroscopic residual stress and the strain response of a single lattice reflection depends on many features including orientation, elastic compliance, anisotropic yield and the constraint provided by surrounding grains. Currently the mechanics of material deformation are not well understood and there is often ambiguity concerning the nature and origin of the strain measured by a single lattice reflection. The current interest in microstrain effects and grain interaction strains can be well served by measurements on a pulsed source. Stress rig and pressure cell experiments are easily accomodated and allow a range of lattice responses to be examined simultaneously.

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