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THE USE OF PULSED NEUTRON DIFFRACTION TO MEASURE STRAIN IN COMPOSITES

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ABSTRACT

Neutron diffraction is a technique for measuring strain in crystalline materials. It is non destructive, phase discriminatory and more penetrating than X rays. Pulsed neutron sources (in contrast with steady state reactor sources) are particularly appropriate for examining heterogeneous materials or for recording the polycrystalline response of all lattice reflections. Several different aspects of composite behavior can be characterized and examples are given of residual strain measurements, strain relaxation during heating, applied loading, and determination of the strain distribution function.

INTRODUCTION

The fact that neutron diffraction, despite its limited availability has achieved a high profile in stress measurement, in general, and in composites in particular, is a testament to the unique nature of the technique. It offers non-destructive measurement of the average phase strain from volumes of a few mm^3 up to several cm^3 . To date much of its exposure in the literature addresses spatially resolved measurements from sampling volumes (typically 1 - 100 mm^3) inside single phase components. When a single lattice reflection is sufficient, spatially resolved measurements are currently more effectively pursued at reactors, with their greater monochromatic neutron flux, than at pulsed sources. However for studies of composite systems in which there are two or more phases of interest, and when spatial resolution is not an issue, pulsed neutron sources are especially valuable.

As the benefits of combining materials are explored, heterogeneous systems are becoming increasingly prevalent and include examples such as metal matrix composites (MMC) like aluminum reinforced with silicon carbide, bonded metal-ceramic laminates and continuous fiber reinforced materials. In most cases the beneficial mechanical properties require a strong bond between phases that have different mechanical characteristics and usually also have different coefficients of thermal expansion (CTE). Thus residual stress between phases is common. Since it affects properties such as strength or fracture toughness as well as influencing the durability, debonding, and damage tolerance of composites [1], knowledge of its origin and magnitude is important.

Strains in composites are intrinsically variable and periodic over length scales comparable to the size of the reinforcing material. Microstructural heterogeneity can vary from μm to mm . To measure average phase strains at the smaller end of this scale, for example in micron sized SiC particles in an MMC, the preferred experimental approach is a diffraction-based technique. Although X-ray diffraction is widely used, it is usually limited to within 100 μm of a surface, which is often unrepresentative of bulk behavior [2]. By contrast, neutrons interact weakly with most materials of engineering interest and offer an alternative more penetrating volume averaged measurement [3].

The most commonly reported measurements are of residual strains but a more comprehensive understanding of composites can be achieved by recording the evolution of phase strains during and as a result of thermo-mechanical loading and by considering the strain distribution about the mean strain value. In this paper the merits of pulsed sources are discussed with examples taken from research using the pulsed neutron source at Los Alamos. In the interest of brevity spatially resolved measurements will not be discussed.

NEUTRON DIFFRACTION FUNDAMENTALS

There are a variety of references [4,5] describing the neutron diffraction method, of which to date the most comprehensive is the proceedings of a 1991 NATO workshop [6]. Consequently, only a brief overview is given here. The use of neutrons to make strain measurements is similar to the widespread use of X rays [7]. When a polycrystalline material is deformed by a stress (residual or applied), the interatomic lattice spacings are altered from their unstressed values. If the yield stress is not

exceeded, the deformation is reversible; otherwise, it includes a plastic component. By recording the change in wavelength of a diffracted peak at a fixed angle, or the change in angle of a diffracted peak at a fixed wavelength, it is possible to measure the *elastic* component of strain in a polycrystal. The scattering vector (\mathbf{Q}), which bisects the incident and diffracted neutron beams, defines the direction in which the strain is measured. At a steady state (reactor) source, measurements are usually made at a fixed wavelength, and the scattering vector for each Bragg reflection falls at a different angle. Thus to measure the strain in the *same* direction in a specimen for multiple Bragg peaks, it must be rotated for each new peak. If only reflections with fortuitous scattering angles are examined, this limits the characterization of the complete polycrystalline behavior.

PULSED NEUTRON SOURCES

At a reactor, a very high flux of neutrons is produced but since most strain measurements are performed at a monochromatic wavelength only a small fraction of that flux is used. A pulsed source, on the other hand, operates in what is called time of flight (TOF) mode and although the time average flux is less, in a diffraction experiment all neutrons in each pulse contribute to the recorded spectrum at a fixed angle. Discrete pulses of neutrons are produced by a process called spallation which occurs when energetic protons interact with a heavy metal target (at LANSCE - 800MeV protons on Tungsten). The neutrons in each pulse constitute a continuous energy spectrum with a distribution determined by the material, temperature and dimensions of a moderator close to the target. Specimens are "scanned in wavelength" and lattice spacings, d_{hkl} are calculated from the wavelengths, λ_{hkl} , corresponding to diffracted peaks at a fixed scattering angle. The wavelengths of detected diffracted neutrons are determined from their "time of flight" between production and detection. After compiling the data from many pulses, diffracted spectra that contain all the Bragg reflections for each phase are produced. Noting that the velocity of a neutron is inversely proportional to its wavelength, to get the elastic lattice strain, from changes in the diffracted wavelength of a reflection at a fixed angle (recorded as a difference in the time of flight), we use:

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

where t_{hkl} is the time of flight for a particular hkl reflection. Strains are determined from changes in time of flight, Δt_{hkl} , for diffracted maxima. Each reflection can be fitted individually to assess the polycrystalline anisotropy, or the pattern can be treated as a whole to assess the average phase response.

For most spatially resolved strain measurements typically one or two Bragg reflections are used because of the need to juggle the neutron wavelength, scattering angle and rotation of the specimen to keep the scattering vector along a specific direction. Since small ($<100\text{mm}^3$) sampling volumes are the norm high count rates are critical, and the high monochromatic flux of a reactor is to be preferred. However for problems in which a complete diffraction pattern is required, from an intensity point of view, the currently operating spallation sources are already competitive with reactors. In composites, it is implicit that more than one phase is of interest (preferably several reflections in each) and for bulk strain measurements small sampling volumes are not required, then pulsed sources are superior.

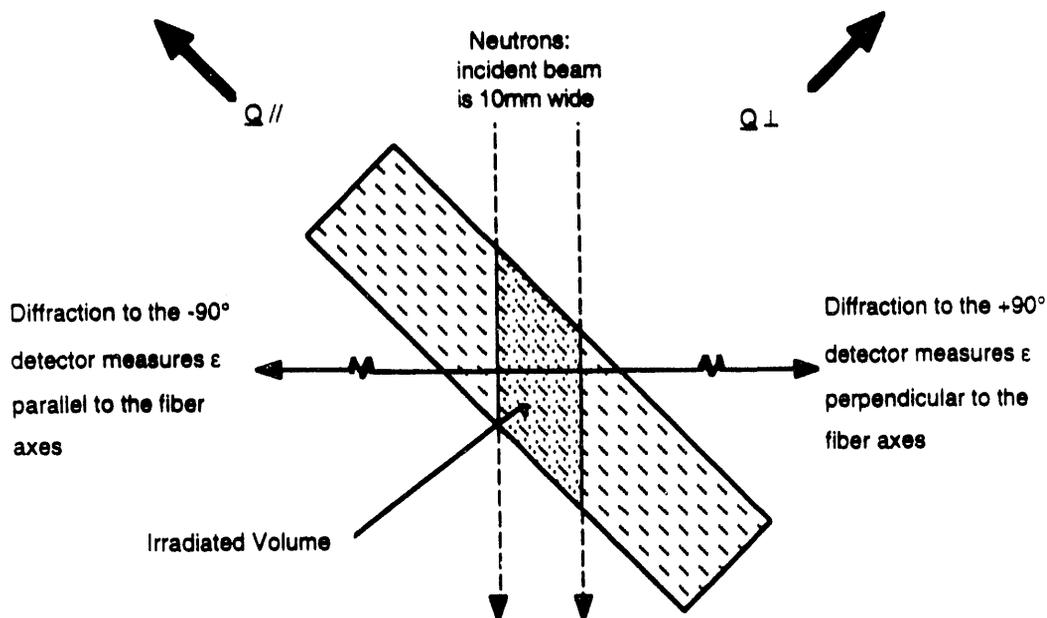


Figure 1. Schematic of specimen orientation in the NPD for a discontinuous aligned fiber composite (the back scattering detectors are not shown)

The advantages associated with a pulsed source derive from the ability to collect a complete diffraction pattern in a detector at a fixed angle. On the Neutron Powder Diffractometer (NPD) at the Manuel Lujan, Jr. Neutron Scattering Center (LANSCE), complete diffraction patterns (thus strains) are recorded simultaneously in 4 directions by 4 detector banks. For example, in the case of an aligned fiber composite opposing 90° detectors simultaneously measure axial and transverse strains (fig 1). In practice each detector subtends an angle typically about $10^\circ 2\theta$ corresponding to a range in Q of 5° from end to end. The best resolution is achieved in the back scattering banks. Detectors placed in any scattering geometry can collect a pattern- which facilitates the use pressure cells or experiments for which a fixed scattering geometry is required. Similarly the penetrability of neutrons facilitates measurements inside a furnace or cryostats.

EXAMPLES

Neutron diffraction provides opportunities for investigating aspects other than simple post fabrication strains. These include; measurement of the temperature at which incompatibility strains begin to be introduced on cooling from fabrication temperatures, the evolution of strain due to applied loads or plastic deformation, and assessment of the strain distribution. Examples for different material systems are listed below.

EXAMPLE 1: RESIDUAL STRAIN IN DEFORMED CUNB

Incompatibility strains in composite systems have been reported for many different systems. Recently the evolution and subsequent directionality of these strains following plastic deformation have been a subject of interest, particularly in silicon carbide reinforced aluminum - SiC/Al [8,9]. This example reports the strain in the Niobium phase of copper niobium specimens that have been subjected to plastic deformation in different temperature and strain rate regimes. By contrast with SiC/Al in which the ceramic reinforcement can be treated as a hard elastic material, CuNb is a co-deforming composite in which both phases can deform plastically. Alloys containing insoluble ductile phases can be heavily worked by rolling or swaging to give microstructural properties similar to those found in conventional lamellar or fibrous composites. These materials are often termed HDISC for heavily deformed *in situ* composites. The ability to co-deform CuNb depends, either on the production of large elastic stresses in the harder Nb phase or the provision of compatibility by the accumulation of substructure, such as dislocations, at the interface. Differing expansion coefficients mean that thermal residual stresses (TRS) already exist before deformation. The residual stresses are significant because they induce changes in the defect density which in turn affect the macroscopic properties. Thus their measurement is important for linking continuum with micromechanical models and for optimizing fabrication methods.

Copper-niobium has a high thermal and electrical conductivity combined with relatively elevated strength. In common with CuAg [10] one potential application is for high strength - high conductivity wires for pulsed magnets. When the current is switched on the coils are subjected to rapid loading due to Lorentz forces. In the presence of such loading the load-strain response of the niobium is highly dependent on temperature and strain rate. Conversely the yield strength of the annealed copper is only weakly dependent on temperature and strain rate. At high strain rates or cold temperatures the niobium behaves like a harder material than at low strain rate or higher temperatures. The examined material consisted of a copper matrix containing 15vol% Nb particles (approximately spherical 4 μm in diameter). Following a 930°C anneal for 1 hour 4 cylinders (original dimensions 10mm diameter, 13mm long) were deformed in compression at two strain rates at room temperature (RT) and low temperature (LT) (see Table 1). Neutron measurements were made of the residual strains. Only data for the Nb parallel to the loading axis are reported (fig. 2). The strains are relative to an undeformed sample of the starting material, thus are not absolute.

Table 1 Temperature and strain rate of CuNb specimens

Sample	1	2	3	4
Temperature K	293	293	77	77
Strain rate s^{-1}	0.001	1000	0.001	1000
Plastic strain	0.1	0.15	0.1	0.15

Parallel to the loading axis, the niobium minority phase is between -2000 and -5500 μstrain in compression depending on the lattice reflection. This is balanced by smaller tensile strains (not shown) in the copper (0 to 400 μstrain). At low temperature or high strain rate the niobium is expected to have the highest yield stress so larger residual stresses were expected and measured. Currently the efficacy of finite element models are being tested against the neutron results.

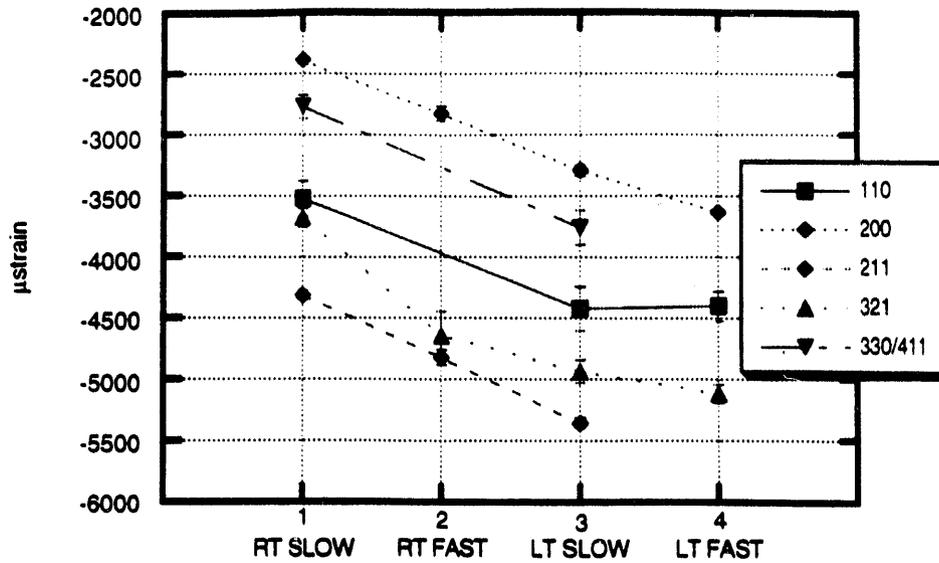


Figure 2 Residual elastic strain in niobium parallel to loading direction following deformation

EXAMPLE 2: STRAIN RELAXATION WITH TEMPERATURE IN A CUSIL- ALUMINA LAMINATE

One significant parameter in modelling composites is the temperature at which strain begins to be developed because of thermal incompatibility. Above this temperature diffusion and creep act to relax the incompatibility strains while below it the magnitude of the induced elastic strains in the two phases depends on the temperature drop, difference in the CTEs and the respective yield stresses. The temperature at which this ceases to be true can be determined using neutron diffraction [12]. When a composite is heated the constituents expand according to their respective CTEs and, simultaneously, the TRS strains are relaxed as the thermal mismatch between phases is reduced. By comparing the temperature dependence of lattice parameters (or individual reflections) in unbonded reference materials with those in the composite it is possible to determine the stress free temperature. Clearly this is the same for both phases.

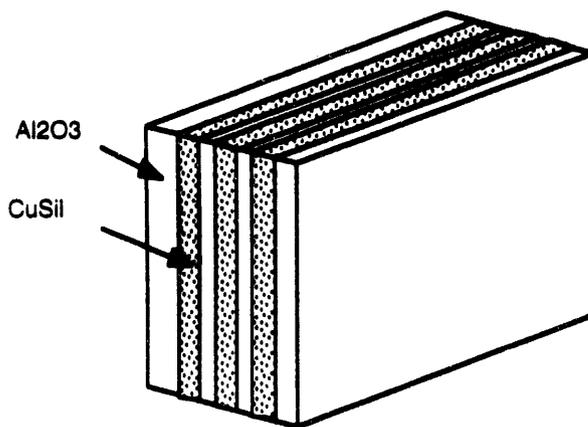


Figure 3a. Schematic of CuSiI(3 layers) Alumina (4 layers) composite

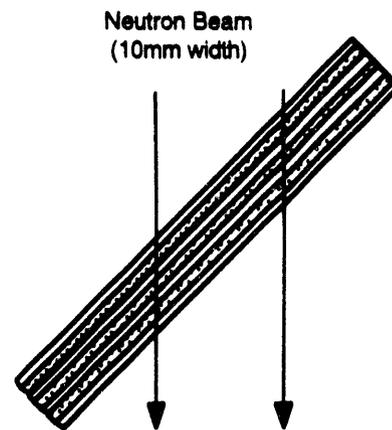


Figure 3b. Plan view of neutron beam incident on specimen at 45°

Multilayered composites consisting of bonded alternating ceramic and metal layers offer attractive structural properties, however TRS are usually present affecting both the integrity of the bond and crack propagation across or along the interface. Rockwell International is studying alumina aluminum multilayer ceramics for a variety of possible applications. Complementary studies on an alumina-Cusil (active braze alloy: 63wt% Ag, 1.75%Ti, balance Cu) system were made because the strains were expected to be larger (easier to measure) due to the higher Cusil yield stress than aluminum. In this example, preliminary results are reported of a heating measurement on a bonded specimen consisting of 4 alumina layers alternating with 3 Cusil layers (fig. 3a). The sample was placed in the beam as shown in figure 3b. The total thickness of the sample was approximately 5mm.

The strain in the in-plane direction of the alumina is expected to be in compression because of the greater CTE of the braze alloy (18ppm /°C) than the ceramic (8ppm /°C). As the multilayer specimen is heated the strain is relaxed until the lattice parameter of the alumina is identical to the lattice parameter of an alumina standard, heated in a separate experiment. By comparing the two, the reduction in elastic compressive strain in the alumina can be calculated as a function of temperature. Figure 4 shows the expansion of the alumina in the laminate and the standard plotted as a strain difference from room temperature. At room temperature the standard is started at 0 strain while the laminate is displaced to -500 μ strain which is the starting residual strain. As the temperature is increased the standard "strain difference" shows a linear increase while the laminate "strain" shows a curved variation. Unlike a simple two phase system in which the expansion of both laminate and reference would be expected to be linear the non linearity of the alumina in the laminate is presumed to be due to the multiphase nature of the braze. The difference between the two lines corresponds to the elastic mismatch strain. The apparent increase in strain between room temperature and about 300°C is not understood. From a rough extrapolation the strain free temperature appears to be about 600°C, compared with the consolidation temperature of 750°C. Clearly more temperatures need to be examined for a better understanding of the temperature dependence. A similar calculation was not done for the braze alloy because it consisted of multiple phases.

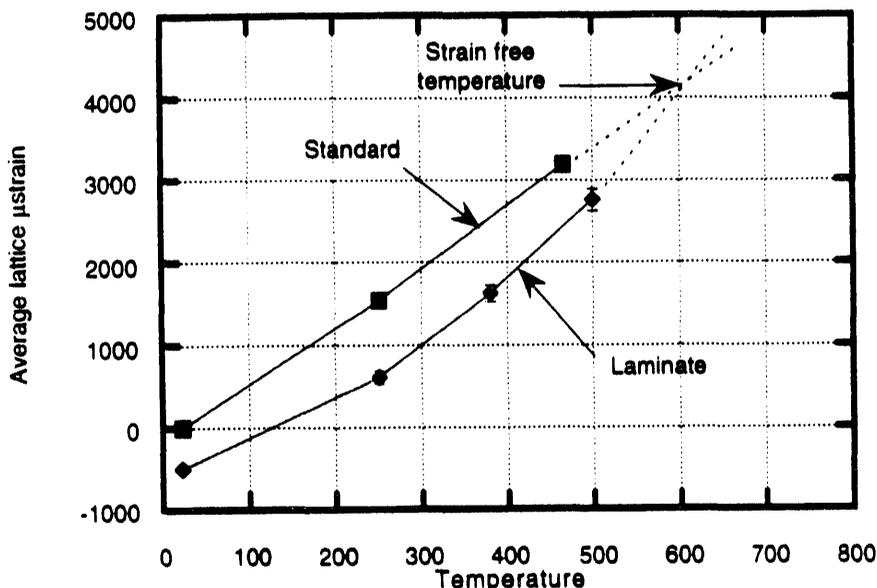


Figure 4 In-plane alumina lattice parameter laminate (4 sheets alumina, 3 sheets Cusil) and standard as a function of temperature (plotted as a strain from room temperature).

EXAMPLE 3: APPLIED LOADING OF ALSIC AT 110°C

Measurement of phase strains during the application of static loads provides insight into the mechanisms and onset of relaxation and load transfer. Diffraction methods measure elastic strains thus non linearity in plots of applied loading vs. elastic strain in individual phases of a composite is indicative of load transfer. Although non-linearity does not provide unambiguous identification of the active mechanisms it does offer a test for material models [13]. Apart from measurement of all the lattice reflections in both phases, the ability to measure strain simultaneously parallel and perpendicular is a strong reason for performing these measurements at a pulsed source since this cannot easily be achieved at a monochromatic source.

Simultaneous high temperature and applied load measurement provide another dimension valuable for characterizing composites that may be expected to operate in temperature regimes above ambient. For example many of the automobile applications of SiCAl such as connecting rods, brake rotors or drive shafts are expected to see sustained or periodic temperature fluctuations. In this example we present strains measured in a uniaxial tension test on a 15 vol% SiC Al material (supplied by DWA). 1" long (125W) cartridge heaters at either end of the sample controlled the center at 110°C (although the ends were hotter). The specimen was surrounded by a vanadium heat shield. The strains parallel to the loading direction are shown in figure 4. For clarity only a few reflections are given. The inset shows the macroscopic strain recorded using a strain gauge.

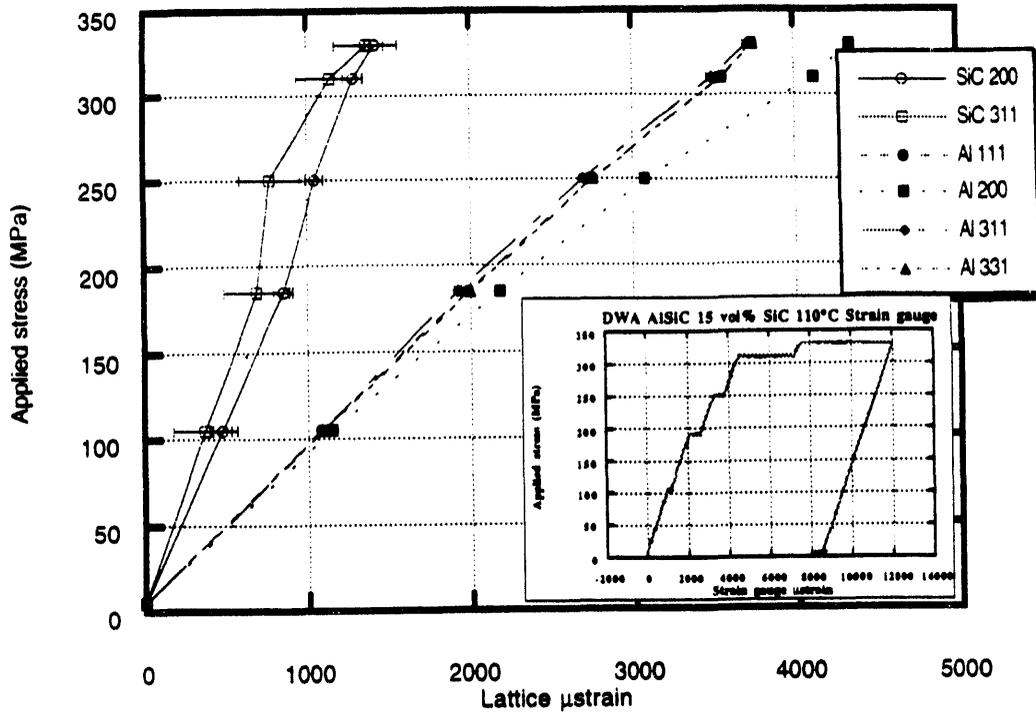


Figure 5 Strain relative to initial material state in a uniaxial tension test of a 15 vol% SiC Al 6091 (T6) particulate MMC.

Each measurement at a static load level took approximately 4 hours. Above 200MPa the sample was creeping over the duration of each stress level. On unload (not shown) the Aluminum reflections were left slightly in tension ($\approx 100\mu\epsilon$) relative to the starting state of the material and the silicon carbide in compression ($< -100\mu\epsilon$). Different reflections showed different residual strains with the largest occurring for the Al 200 which was close to $500\mu\epsilon$, evidence for which can be seen in figure 5 where it bends away from the 3 other aluminum reflections. The sign of the residual strains suggests that diffusional rather than plastic relaxation of the aluminum occurred. Providing that the morphology of the loading curves can be identified with sufficient accuracy inferences about the material deformation can be made [14].

Although there is a disparity in the time available for neutron diffraction measurements and typical creep tests, nevertheless the ability to make measurements under load and at temperature opens possibilities of recording stress redistribution. At the end of 1993 at LANSCE uniaxial tension tests were made at 400°C. With an improved furnace applied loading measurements could be made at much higher temperatures. Equally there is the possibility of studying combinations of stress and/or temperature induced phase transformations in a variety of material systems.

EXAMPLE 4 MEAN INTERNAL STRAIN DISTRIBUTION FUNCTION IN Al_2O_3 SiC

The strains reported in the previous examples were all calculated from the displacement (in time of flight) of the peak center. The peak shape used to fit reflections at the pulsed source at LANSCE is a Gaussian convoluted with rising and falling exponentials. This is a reasonable description of the peak shape (associated with the production neutrons in the moderator with each pulse). In reality the shape of a Bragg reflection is given by a convolution of the instrumental resolution with a peak

shape determined by microstructural and strain broadening effects. Thus although for powders the assumption of a Gaussian is reasonable, in a solid it may not be. In general, uncertainty about microstructural or particle size broadening precludes an analysis of the peak shape to give the strain distribution but in specific cases if the modification of a peak shape can be attributed to strain effects alone then it is possible. In its simplest form this is achieved by comparing the widths of a strain free reference peaks with the corresponding widths in the composite.

Recognizing that it should be possible to extract a volume averaged strain distribution function Todd and Derby described an approach for doing so [15]. The method requires that composite and reference peaks are measured on the same instrument in the same geometry (thus have the same instrumental resolution function) and that neither particle size nor microstructural broadening effects apply. Then the broadening of a composite peak is a convolution of a reference peak with a function that depends only on the strain distribution. This function is called the mean internal distribution function (MISDF). It describes the probability of a fraction of the material in the gauge volume having a strain in a given range and can be solved for by using Fourier transform techniques.

To illustrate this approach the MISDF is presented for a single alumina peak in a discontinuously reinforced 30vol% silicon carbide alumina composite. The average compressive stress in the SiC was about 1GPa. By smoothing and deconvoluting the measured peaks, the MISDF for the alumina 113 peak was calculated and is shown for data taken on two different instruments in figure 6. The two neutron diffractometers had significantly different resolutions, HRPD at ISIS ($\Delta d/d \approx 7 \cdot 10^{-4}$) and the NPD at LANSCE ($\Delta d/d \approx 1.5 \cdot 10^{-4}$). Although the average alumina strain is tensile (as would be determined only from the peak centers) there is a tail to the distribution showing that a significant fraction is in compression. This is not surprising because the measurement is of the strain in a single direction throughout the material and for example the strain in the same direction will differ around an included particle.

The ultimate goal of a strain measurement in a composite is to resolve the strain distribution across the reinforcement / matrix interface. Although the MISDF can not do this, in those cases when it is applicable it can provide a volume averaged probability distribution of strain against which models can be tested. The relevance of not only the mean strain values but also their distributions, to mechanical properties have not yet been clearly identified. For example sharp angular particles are more likely to have extremes of strain associated with their vertices and this presumably has implications to their integrity. Since there are many reflections to choose from in a typical diffraction pattern it is possible to check the validity of the MISDF's derived. One problem is that the technique does require clean reflections i.e., no overlapping reflections from other phases and for composites consisting of two low symmetry crystal structures the degree of overlap can be considerable, particularly at short d spacings, making it hard to find suitable candidate reflections.

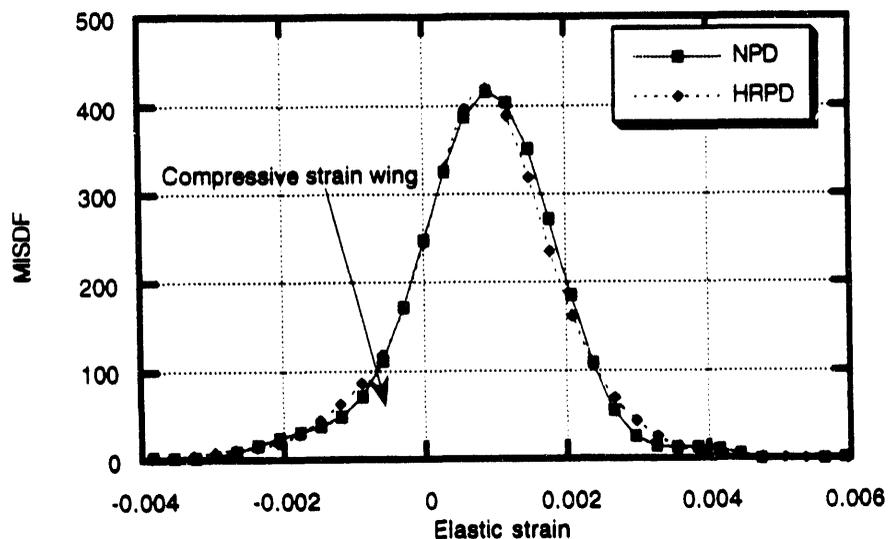


Figure 6. MISDF for the Al_2O_3 113 reflection in a discontinuously reinforced alumina silicon carbide composite. Measured at LANSCE and ISIS with instruments of 30m and 100m flight paths respectively.

RELEVANCE OF NEUTRON MEASUREMENTS TO NUMERICAL CALCULATIONS

The strength of diffraction techniques lies in their ability to separate average phase-strains, providing a measurement of stress partitioning which is one of the basic phenomena in composites. Phase-strains obtained from changes in the position of a Bragg reflection, correspond to a volume average over many grains in which the measured crystallographic planes are

perpendicular to the diffraction vector. Of course the strain field outside and inside real particles varies, with steep gradients. Thus although a measurement can distinguish between phases it does not resolve the true strain distribution around particles merely a volume average of it. Composite applications often rely on accurate predictions of mechanical properties such as strength, fracture toughness, durability, debonding, or damage tolerance. These predictions usually require knowledge of the distributions of field quantities across phase boundaries which are often estimated using finite element codes.

Typically in finite element models one particle is embedded in a matrix of surrounding material which is assumed to repeat indefinitely. Then constitutive modeling of complex loading paths is possible. To be effective, the code must satisfactorily describe the stress distribution between constituents but validation is important because of the variety of physical processes that can occur including particle fracture, interface decohesion and plastic or diffusional relaxation. By taking volume averages over each phase of the pertinent field quantities, in this case strain, comparison with neutron diffraction measurements is possible.

Despite the dilution of the information in a finite element model required for comparison with neutron results, the comparison can still probe materials at the scale of the modeled unit cell. In figure 7 the measured strain perpendicular to the loading direction of an AlTiAl composite is displayed with a finite element prediction for each phase [16]. The lattice phase-strain evolution displays a "zig-zag" increase for both the measured and calculated curves. It was found that if the initial thermal residual stresses were not included in the model, a different morphology was predicted. On examination of the effective strain contours in the FE model it was shown that the presence of the TRS alters the strain (stress) field so that the site of matrix initial yielding changes which in turn alters the morphology of the loading curve. Thus comparison of the experimentally determined neutron volume average with the FEM cell volume average is an indirect tool for probing the localized strain behavior.

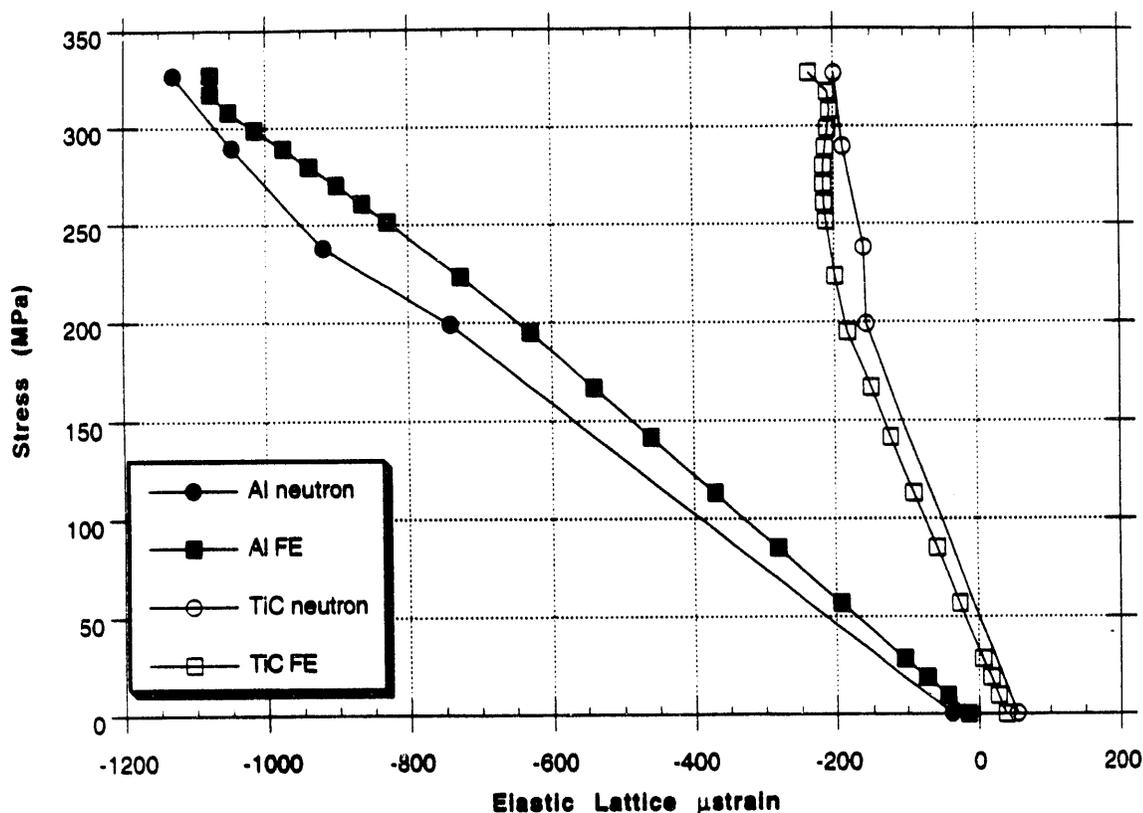


Figure 7 Measured strains perpendicular to the loading direction of a uniaxial tension test in an Al 2219 15vol%TiC MMC with corresponding FE predictions.

One issue that has not been sufficiently addressed in comparing diffraction results with numerical models is the significance of the strain anisotropy associated with different crystallographic planes. This contrasts with continuum modeling approaches in which the materials are assumed to be smooth. Although a qualitative assessment of the collective phase behavior of all lattice reflections is possible by performing a Rietveld refinement [17] it is not a quantitatively valid approach particularly in cases of severe texture or plasticity.

DISCUSSION

It is hard to generalize about what experiments are possible at pulsed sources since count times are specimen, instrument and source dependent. For example using the NPD at LANSCE, which is the highest resolution spectrometer in the US, spectra are attained in each of the surrounding detectors from $\approx 1000\text{mm}^3$ of aluminum silicon carbide after about 4 hours. The strains calculated from individual reflections of each phase can then be specified to an accuracy of about $\pm 50\mu\epsilon$ (suitable for measuring strains in stiff ceramics). Larger sampling volumes, stronger scattering materials or shorter flight path spectrometers (greater intensity at the expense of poorer resolution) are all factors which shorten the count times. Currently there are 4 operating pulsed neutron sources which are, in order of source flux, ISIS in the UK, LANSCE at Los Alamos, IPNS at Chicago or KENS in Japan

One problem specific to a pulsed source is the need to ensure that specimens and their corresponding standards are placed in the same position and geometry. Since the elastic strains of interest are usually less than 0.5% even small differences in position of the reference with the composite can change the flight path and be misinterpreted as a strain. Indeed identifying a suitable unstrained reference in itself is often difficult. While relative strain changes within existing materials are relatively easy to measure, determination of absolute strains requires a standard of the same stoichiometry, microstructure and texture as the specimen. Particularly in composites in which the material microstructure is intimately related to the production and strain state associated with the composite itself this is extremely difficult to achieve.

One practical bottleneck at present pulsed sources is handling and using all the data. Fitting all of the available diffraction peaks in each pattern is labor intensive even if automated. Typically only combinations of cubic materials have many non-overlapped reflections while low symmetry crystal composites have considerable overlap particularly at short d spacings. Identification of individual peaks is often time consuming, particularly if there is a variable texture or during a heating experiment in which peaks move by different amounts sometimes causing smaller previously unresolved reflections to appear in the wings. Since the strains of interest usually correspond to peak shifts considerably less than the peak half widths it is important to check visually that each fit is clean and uncontaminated by neighboring peaks. If more powerful pulsed sources are constructed serious consideration of the data analysis issues will be needed to allow real time data analysis.

Having fit the data, interpretation of the sometimes highly anisotropic crystalline response is the next problem. Qualitative interpretation and simple averaging to give an average response is often sufficient for comparing with continuum models, but it is becoming apparent that more quantitative treatments for relating polycrystalline response to macroscopic properties are needed. For example the textural state of aluminum is known to play a significant role in drawing processes. Although the implications of texture to diffraction strain measurements have been discussed [18], routinely relating anisotropic polycrystalline strain effects and texture to continuum mechanics or engineering models remains a challenge. To meet it collaboration is needed between researchers describing plasticity, 3D texture evolution and polycrystalline modelling and researchers performing this type of neutron strain measurements since the former provide the tools to understand the latter and both are needed to provide an overall interpretation.

CONCLUSION

Pulsed neutron diffraction is a technique for measuring the introduction, presence, evolution and distribution of strain in crystalline composites. It gives a measure of the volume average phase strain thus providing information about stress partitioning while simultaneously recording texture and polycrystalline anisotropy. Multiple strain directions can be examined simultaneously. Most commonly strain is interpreted from the change in wavelength of diffracted reflections at a fixed angle, but in ideal cases a MISDF can be extracted by analysis of the peak shapes. By combining several avenues of neutron investigation a comprehensive assessment of the composite strain evolution can be obtained and compared with numerical calculations.

ACKNOWLEDGEMENTS

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