

Conf. 92-3596-2

LA-UR- 92-3596

Title: Strain Measurement in Individual Phases of an Al/TiC Composite During Mechanical Loading

LA-UR--92-3596

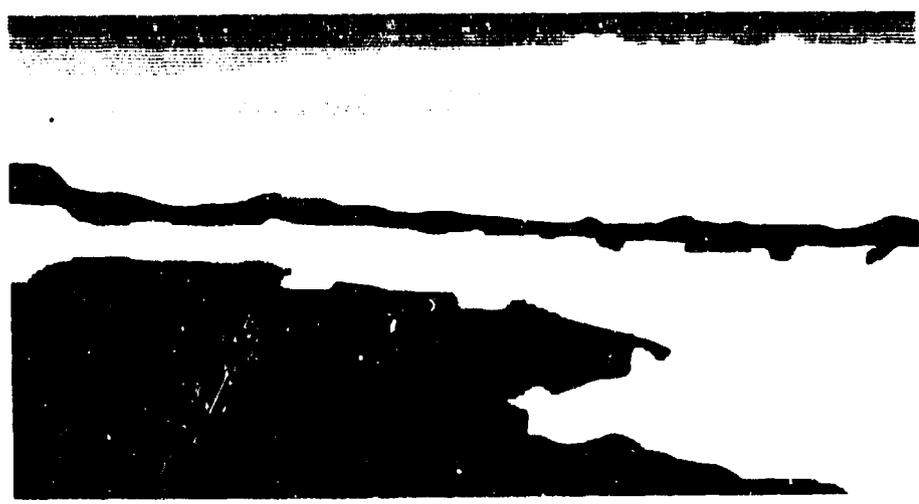
DE93 003806

DEC 0 1992

Author(s): M. A. M. Bourke, LANSCE
J. A. Goldstone, LANSCE
M. G. Stout, MST-5
A. C. Lawson, MST-5
J. E. Allison, Ford Motor Company

Submitted to: Annual TMS Meeting, February 21-25, 1993, Denver Colorado

MASTER



This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Los Alamos
NATIONAL LABORATORY

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the University of California for the U.S. Department of Energy under contract W-7405-ENG-36. By acceptance of this article, the publisher recognizes that the U.S. Government retains a nonexclusive, royalty free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes. The Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy.

**Strain measurement in individual phases of an Al/TiC
composite during mechanical loading**

M. A. M. Bourke¹, J. A. Goldstone¹, M. G. Stout², A.C. Lawson²,
and J. E. Allison³

¹ Manuel Lujan Jr. Neutron Scattering Center, Los Alamos National
Laboratory, Los Alamos, NM, 87545, USA

² Materials Science and Technology Division, Los Alamos National
Laboratory, Los Alamos, NM, 87545, USA

³ Scientific Research Laboratory, Ford Motor Company,
Dearborn, MI 48121-2053, USA

Abstract

The macroscopic and microscopic stress states of metal matrix composites depend on their fabrication and deformation history. The situation is complex because in addition to crystalline anisotropies, physical processes like fiber breakage and plastic or diffusional relaxation may interact during production and service. Numerical codes are frequently used to predict the development of residual strains as a result of such processes: the complexity of the situation makes experimental validation important during and after thermo-mechanical conditions that simulate service. Neutron diffraction provides a unique method for examining materials during thermo-mechanical loading because it is nondestructive and penetrating and can distinguish between the strains in individual phases. Using a pulsed neutron source, all lattice reflections are recorded in all constituents simultaneously. Preliminary *in-situ* strain measurements under load of an aluminum titanium-carbide composite are presented here. The measurements were made using a compact stress rig on the neutron powder diffractometer at the Manuel Lujan Jr. Neutron Scattering Center at Los Alamos National Laboratory.

Introduction

In metal matrix composites the presence of a hard minority phase can promote localized or non-homogeneous plastic flow during loading. This leads to internal elastic strains that are superimposed on initial strains due to thermal incompatibilities. Even in single phase polycrystalline materials, strain incompatibility and directionally dependent yield stresses in individual grains can lead to strain differences in adjacent grains following plastic deformation (1). In some cases, microstrain effects can result in some grains experiencing a tensile residual stress even when the macroscopic stress is compressive. This may affect crack initiation and is likely to affect mechanical properties such as strength and fracture toughness.

In a metal-matrix composite (MMC), the situation is even more complex because processes distinct from crystalline anisotropy may be contributing (2). Numerical codes are frequently used to predict the development of residual strains as a result of such processes, but the complexity of the situation increases the importance of experimental validation during and after thermo-mechanical conditions that simulate service. Neutrons have already been used and identified as a valuable method for validation of numerical codes (3,4). Measurements have largely concentrated on residual stresses following heat treatments or deformation. By making measurements during the application of a load, we can improve our understanding of the performance of MMCs during yield.

Neutron diffraction measurement

Diffraction methods of measuring strain by x-rays or neutrons have been extensively covered in the literature (5-9) and only a brief overview is given here. Changes in the lattice spacings of crystalline materials experiencing a residual or applied load are the basis of strain measurement by diffraction. When x-rays or neutrons of an appropriate wavelength fall on a polycrystalline material, diffraction peaks are produced corresponding to the spacings of atoms within the material. Bragg's Law relates the lattice spacings to the angle and wavelength of a diffracted x-ray or neutron. If these values are known, the lattice spacing for a set of crystals in specific orientations can be determined. The direction in which strain is measured lies along the scattering vector (Q) and is dictated by the scattering geometry to the detector.

In contrast with x-rays whose penetration is limited to surfaces, neutrons can penetrate several tens of millimeters into most materials of engineering interest. The low attenuation enables many grains to be examined, giving a representative value of the internal strains in grains

of particular orientations. Elastic strains are determined from changes in lattice spacings from a "stress-free" value. For measurements under load, if the unloaded state is used as a stress-free value it must be noted that the initial stress state may include a significant contribution of residual stress from fabrication. The strains of interest are usually less than 2×10^{-3} . Particularly for the ceramic reinforcement residual strains are often small and high-resolution instruments are needed to discern them.

Two types of neutron sources exist, reactors and spallation. In contrast with a reactor source, where monochromatic or single wavelength neutrons are used, at spallation sources pulses of neutrons are used. The wavelength of a detected neutron is determined from its time of flight between creation by the spallation process and arrival at the detector. Thus the specimen is scanned very rapidly in wavelength and after many pulses gives a spectrum containing all the lattice spacings. Determination of all the lattice spacings gives a comprehensive measurement of the microstructural stress state. Multiphase problems are usually handled more effectively using pulsed rather than monochromatic neutrons. For this reason composite problems are more practically approached using spallation rather than reactor sources.

The neutron powder diffractometer (NPD) at the Manuel Lujan Jr. Neutron Scattering Center (LANSCE) is the highest resolution spectrometer of its type in the United States and is particularly appropriate for this work. On the NPD a favorable diffraction geometry offers simultaneous strain measurement in three directions. The orientation of a specimen relative to the neutron beam is shown in figure 1. The loading axis is horizontal and at 45° to the incident beam allowing simultaneous axial and transverse strain measurements to be made in opposing 90° detector banks. The advantages of neutrons in general and of pulsed neutrons in particular are summarized in table I.

Table I Advantages and Benefits of using Neutrons

| Advantages of Neutrons |
|---|
| Penetration to tens of mm; 10,000 greater in iron than x-rays |
| Separates the strains in the different phases |
| Nondestructive |
| Selective of crystalline orientation |
| Representative average of bulk behavior. |
| Benefits of Pulsed Neutrons |
| Simultaneous strain in multiple directions |
| Complete diffraction pattern |
| All reflections measure in one specimen direction |

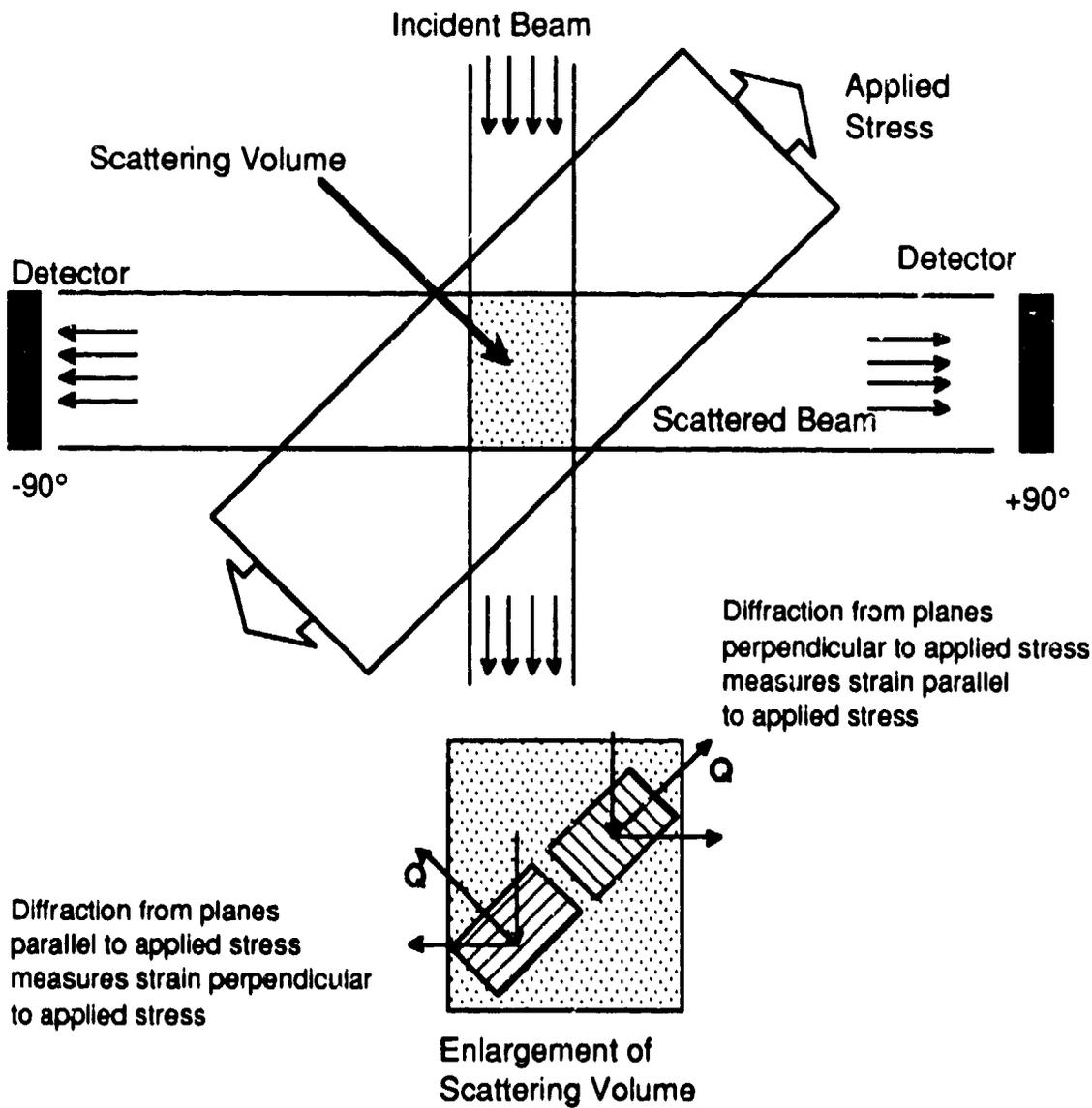


Figure 1 - The experimental setup for *in-situ* strain measurements. Axial and transverse elastic strains resulting from the applied load are measured simultaneously in +90° and -90° detector banks, respectively.

Stress Rigs using neutrons

Stress rigs have been used at reactor sources (10,11) but it is only with the advent of relatively intense pulsed neutron sources like LANSCE, IPNS at Argonne National Laboratory, or ISIS in the UK that comprehensive assessment of strains in composite materials has become possible. Spectrometers at pulsed sources inevitably require more shielding than reactors because the former have more high energy neutrons and gamma rays. This background necessitates more shielding around the sample position and has been the inhibiting feature in developing a stress rig at most pulsed neutron sources. For the NPD the

loading apparatus and frame had to fit into a cylindrical space with an ID of 0.74 m. This precluded any commercial system and necessitated a design in which the actuator was in parallel with the specimen and the load was transferred to the specimen through a pivot arm (figure 2). Care was taken to ensure that the loading on the sample remained axial.

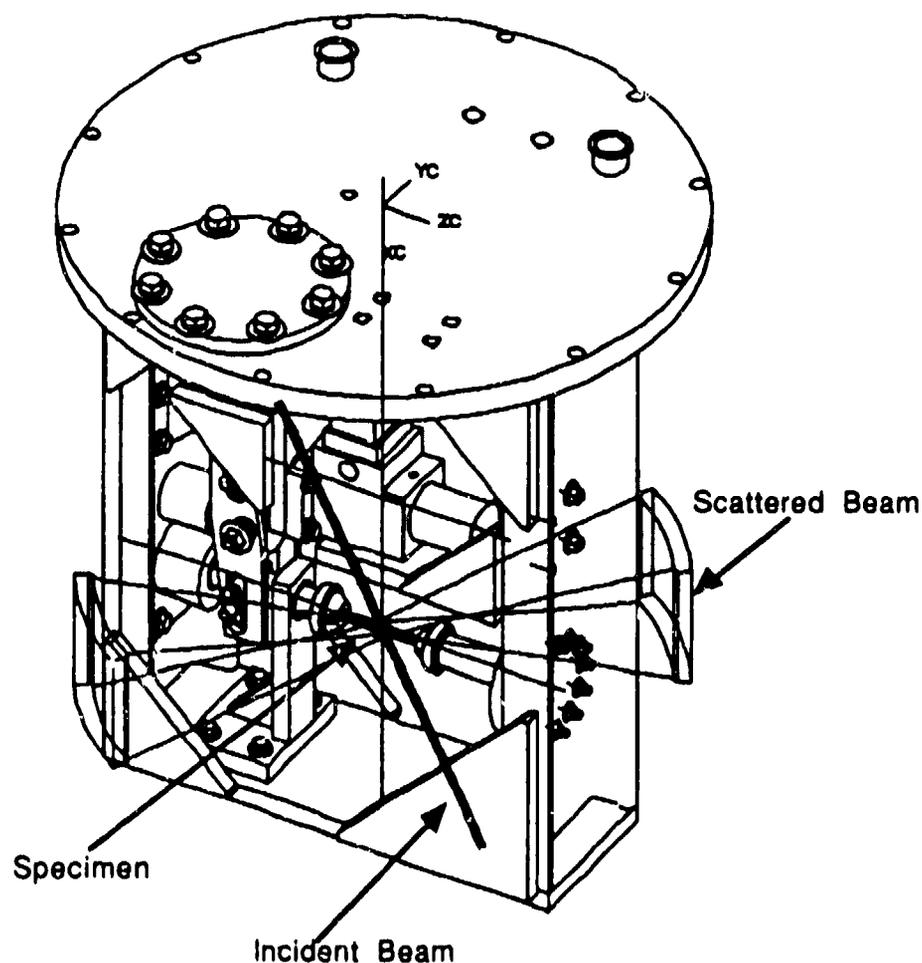


Figure 2 - To operate a stress rig in the limited volume available on the NPD careful design (undertaken by LANSCE designer Dan Davis) was needed to ensure that the incident and diffracted beams were unimpeded.

The characteristics of the loading apparatus that we have made for the NPD are given in table II.

Table II Specifications NPD stress rig

| | |
|--------------------|---|
| Control: | Load or Stroke |
| Max. Load: | ~50kN, Uniaxial tension or compression |
| Sample Size: | 50 to 150 mm in length |
| Sample geometry: | Cylindrical: Grips for different geometries could be made |
| Irradiated volume: | Center 15mm (10mm diameter specimen ~1000mm ³) |
| Temperature: | I Infra red heaters to ~400° C II Planned 1000° C furnace |
| Environment: | Atmospheric, Inert gas, vacuum |
| Control System: | Instron, includes fatigue capability |
| Count times: | 1-12 hours / load level (depending on the material, sampling volume and beam reliability) |

Experimental Measurements

Ford has been investigating a variety of metal matrix composite systems for use in the automotive industry. However most of these potential components are subjected to cyclic thermo-mechanical loading and an understanding of the factors influencing their mechanical behavior is needed to ensure their durability. The material used in this study was an aluminum alloy reinforced with TiC particles formed *in-situ* using a process developed by Martin Marietta Laboratories under the tradename, XD. Although Al/TiC is unlikely to be used in production, the TiC particles produced via the XD process have a number of technological and theoretical advantages. They are spherical, possess clean matrix/reinforcement interfaces and do not appear to fracture under either monotonic or cyclic loading.

Although the residual stress state in Al/TiC can be significantly altered by plastic strains of 0.01 or greater the influence of strains less than 0.001 is unknown. A series of residual strain measurements to investigate the effects of small strains and cyclic loading were made in Al/TiC at LANSCE this summer and will be reported elsewhere. To complement these measurements, a limited selection of experiments on the strain during loading are reported here.

The material that we examined was 15vol% TiC in an artificially aged Al2219 alloy. A cylindrical specimen (10mm diameter) was loaded in uniaxial tension. The loading sequence involved measurements at the following static loads:

0-50-100-148-198 - 0 - 199-238-290-327 - 201-102-0 MPa.

The initial sequence was selected to keep the specimen in the elastic region and was followed by an increase in load to give 1% total strain at 327 MPa. On removal of the final load the specimen was left with a permanent strain of $\approx 0.67\%$. The hold duration at each stress level was typically 4 hours but in a few cases was much longer because of poor beam reliability. A typical diffraction pattern is included in figure 3. (Note many more reflections were observable in the region between 4 and 8 nm, not shown in the figure.)

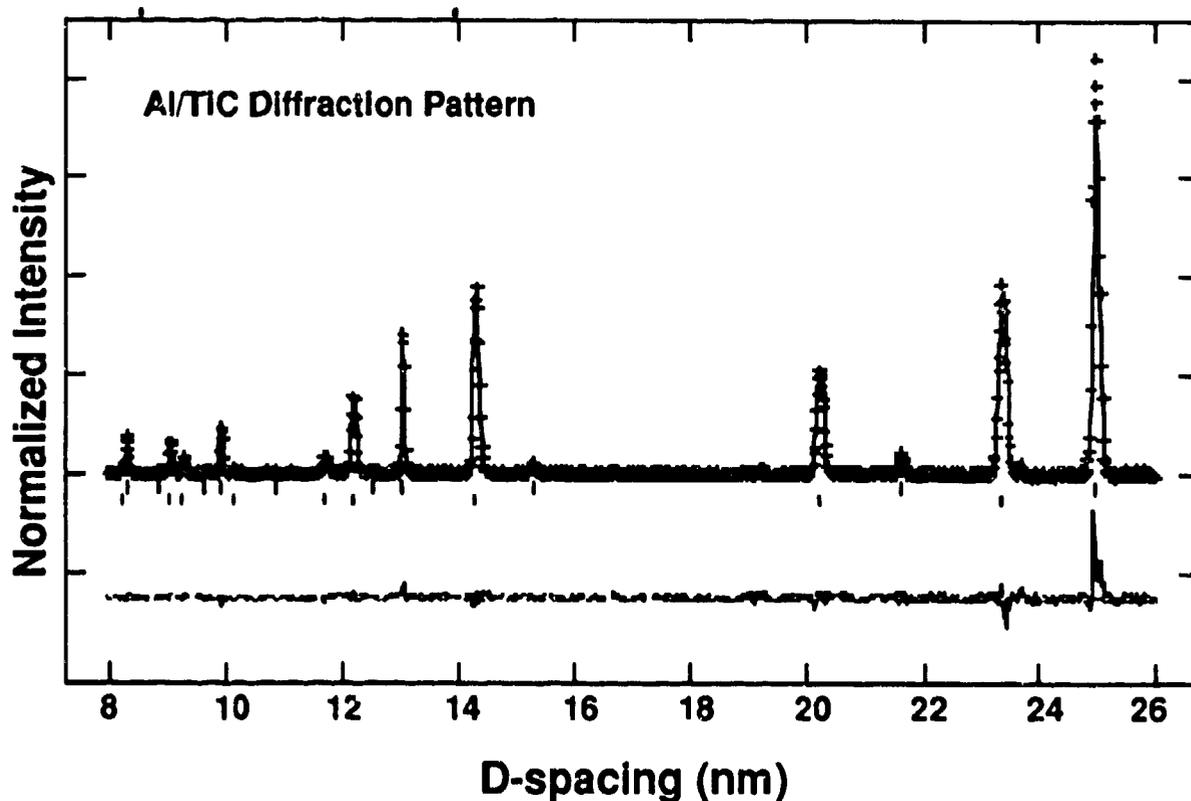


Figure 3 - Diffraction pattern for aluminum titanium-carbide. The pluses are the raw data; the line through the points is the fit to the data; the lower line is the difference between the fit and the data. The tick marks just below the data are the reflection positions for aluminum (lower marks) and titanium-carbide (upper marks). Note: 10 nm = 1 Å.

Bragg reflections in each spectrum can be fitted individually to give strains for grains in different orientations. 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. In regions of compression or tension changes in the lattice parameter can be used to infer the strain.

The use of Rietveld profile refinement provides a quick assessment of the average material state and is appropriate for composites where the

state of stress is well defined in the two phases. However, it should be noted that anisotropic effects do occur for different reflections and the profile refinement will not account for any deviation from perfect crystalline behavior thus is necessarily an approximation. Nevertheless in the absence of severe preferred orientation, the lattice parameter deduced from a Rietveld refinement can (arguably) be assumed to offer a first estimate of an isotropic strain for engineering calculations. (What it fails to do is take any account of crystalline anisotropy which can lead to problems when the assumption of a simple cubic structure starts to break down.)

In figures 4 and 5, we present data showing the strain in the two phases during the applied load as calculated by profile refinement using the lattice parameters and by individual peak fits of selected strong lattice reflections. All of the strains shown are relative to the initial stress state and do not include any contribution due to initial thermal stresses. It should be noted that at the time of press the load cell used in the measurements was out of calibration, so the stresses may change by a few percent before final publication.

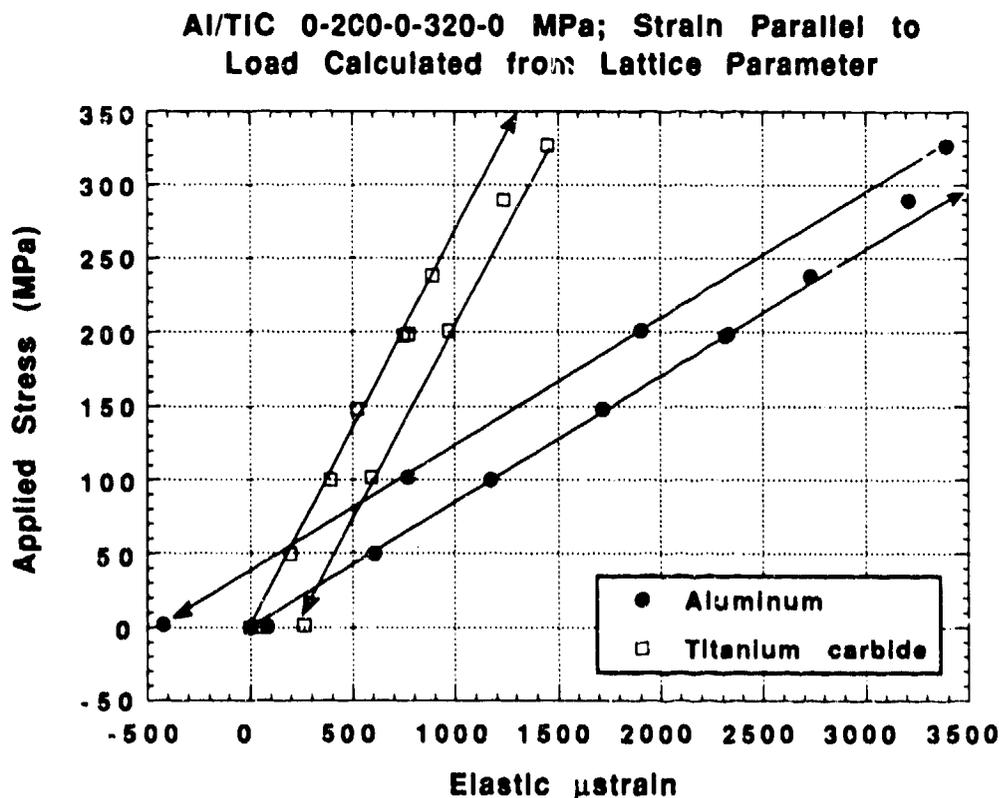


Figure 4 - Strains parallel to the loading direction calculated using the lattice parameter from Rietveld profile refinement. Error bars are approximately the size of the data points.

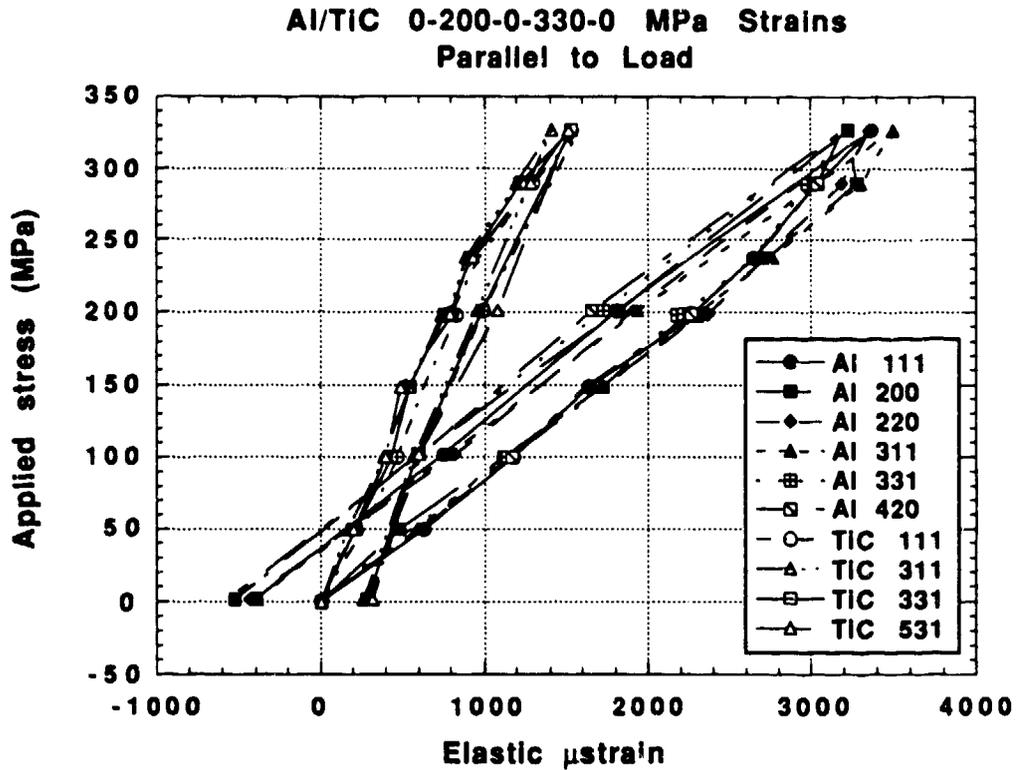


Figure 5 - Strains parallel to the loading direction calculated using selected strong individual lattice reflections.

Discussion

In figure 4, the strain behaviors of the aluminum and titanium carbide remain elastic until 200MPa at which point some deviation from the purely elastic lines can be seen. On unloading significant residual strains (relative to the initial stress state) parallel to the loading directions are induced in the aluminum matrix and titanium carbide particles. The aluminum is significantly in compression and the TiC in tension by about -500 and +250 μ strain respectively. In figure 5, the behavior of the individual lattice reflections clearly mimics the profile refinement results, but anisotropic yield stresses can be seen for the different lattice reflections.

Summary and goals.

To predict the residual stresses that are introduced in MMC systems it is necessary to understand the deformation at a microstructural level. In particular we need to know how and when the individual component

materials deform. In developing a stress rig for operation on the Neutron Powder Diffractometer at LANSCE our objectives were:

To improve our understanding of elastic and plastic deformation of metal matrix composites

and

To provide information required to validate numerical codes.

A pulsed neutron source is powerful for this study because it simultaneously gives strain measurements for all lattice reflections in both phases of an MMC in at least two directions.

To date we have completed measurements on Al/TiC, Al/SiC and Steel and preliminary results on Al/TiC are reported here.

Constitutive modeling of MMC materials following complex loading paths is difficult, but comparisons of experimental data using the stress rig with theory are now possible. With a furnace we hope to be able to examine the onset of relaxation and creep processes at a microstructural level. Our immediate goals are:

To relate these results to residual strain measurements

To compare with modeled predictions

To investigate the effects of plasticity on the diffracted peak widths

To develop a high temperature capabilities measurements in two regimes: 20-400° C and 400-1000° C

To develop specimen grips for sheet and hourglass (reverse loading tests).

To try an *in-situ* fatigue measurement

References

1. S. R. MacEwen, J. Faber, and P. L. Turner, "The use of time of flight neutron diffraction to study grain interaction stresses," Acta Met., 31 (1983), 657-676.
2. A. Allen, M. Bourke, S. Dawes, M. Hutchings, and P. Withers, "The Analysis of Internal Strains Measured by Neutron Diffraction in Al/SiC Metal Matrix Composites," Acta metall. mater., 40, (1992), 2361-2373.
3. G. L. Povirk, M. G. Stout, M. A. M. Bourke, J. A. Goldstone, A. C. Lawson, M. Lovato, S. R. MacEwen, S. R. Nutt and A. Needleman, "Mechanically

Induced Residual Strains in Al/SiC Composites," Scripta Metall. Mater. 25 (1991), 1883-1888.

4. G. L. Povirk, M. G. Stout, M. Bourke, J. A. Goldstone, A. C. Lawson, M. Lovato, S. R. MacEwen, S. R. Nutt and A. Needleman, "Thermally and Mechanically Induced Residual Strains in Al-SiC Composites," Acta metall. mater. 40, (1992), 2391-2412.
5. A. J. Allen, M. T. Hutchings, C. G. Windsor and C. Andreanni, "Neutron diffraction methods for the study of residual stress fields," Advances in Physics, 34, (1985), 445-473.
6. I. C. Noyan and J. B. Cohen, Residual Stress --- Measurement by Diffraction and Interpretation (New York, NY: Springer Verlag, 1987).
7. M. James, M. Bourke, J. A. Goldstone, and A. C. Lawson, "Residual stress measurements in continuous fiber titanium matrix composites," Advances in X Ray Analysis. (New York, NY: Plenum Press, 1992, in press)
8. M. A. M. Bourke, J. A. Goldstone, and T. M. Holden, "Residual stress measurement using the pulsed neutron source at LANSCE," in Measurement of Residual and Applied Stress Using Neutron Diffraction, (ed. M. T. Hutchings and A. Krawitz, Netherlands: Kluwer Acad. Pub., 1992), 369-382.
9. A. Majumdar, J. P. Singh, D. Kupperman, and A. D. Krawitz, "Application of Neutron Diffraction to Measure Residual Strains in Various Engineering Composite Materials," Journal of Eng. Mat. and Tech., 113, (1991), 51-59.
10. A. J. Allen, M. A. Bourke, W. I. F. David, S. Dawes, M. T. Hutchings, A. D. Krawitz, and C. G. Windsor, "Effects of elastic anisotropy on the lattice strains in polycrystalline metals and composites measured by neutron diffraction," Proceedings of ICRES2, (London, UK: Elsevier, 1988) 78.
11. T. Lorentzen and N. Sorensen, "A new device for in-situ loading of samples during neutron diffraction strain measurements," Proc. 12th Risø Int. Symp. on Mat. Sci., 489-496.