Measurement of Strain in Individual Phases of Composites Before, During, and After Mechanical Loading

Joyce A. Goldstone, M. A. M. Bourke, Dan Davis, LANSCE; Ning Shi, LANSCE/CMS; A. C. Lawson, MST-5; Los Alamos National Laboratory, Los Alamos, NM 87545 USA
and
John E. Allison, Ford Motor Company, Dearborn MI, USA

At LANSCE, we have measured residual strains in a variety of metal-matrix systems using the Neutron Powder Diffractometer. In 1992, we developed a compact stress rig for *in-situ* strain measurement during applied loading. Comparison of experimental measurements made with the stress rig on aluminum titanium carbide with numerical predictions showed the importance of the influence of initial thermal residual strains in composites In Al/TiC, normal to the loading direction, the increase in the average matrix and reinforcement lattice strains was in a "zigzag" manner. Finite element modeling indicated that this resulted from residual strains induced during cooling from fabrication temperatures. Data and modeling are presented herein.

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 expansion mismatch. 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 before, 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 loading.

Experimental Method:

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 the diffracted

radiation. 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 and is dictated by the scattering geometry.

In contrast with x-rays, whose penetration is limited, 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 elastic internal strains in grains of particular orientations. Strains are determined from changes in lattice spacings from their "stress-free" values. For measurements under load, if the unloaded state is used as a "stress-free" value, it must be noted that the initial stress state will include residual stresses from fabrication. The strains of interest are usually less than $2x10^{-3}$. Particularly for the ceramic reinforcement residual strains are often less than 10^{-3} and high-resolution instruments are needed to discern them.

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 loading axis is horizontal and at 45º to the incident beam allowing simultaneous axial and transverse strain measurements to be made in opposing 90º detector banks.

Stress Rig:

Stress rigs have been used at reactor [2,10,11] sources 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 or any crystalline multiphase system has become possible. Spectrometers at pulsed sources 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 forced 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 1]. Care was taken to ensure that the loading on the sample remained axial.

Table I 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 14mm [10mm diameter specimen »1000mm^3]
Temperature:	I Cartridge Heaters to »300º C II Planned 1000º C furnace
Environment:	Atmospheric, Inert gas, vacuum
Control System:	Instron, includes fatigue capability, Macintosh with LabView 2™
Count times:	1/2-12 hours / load level [depending on the material, sampling volume and beam reliability]

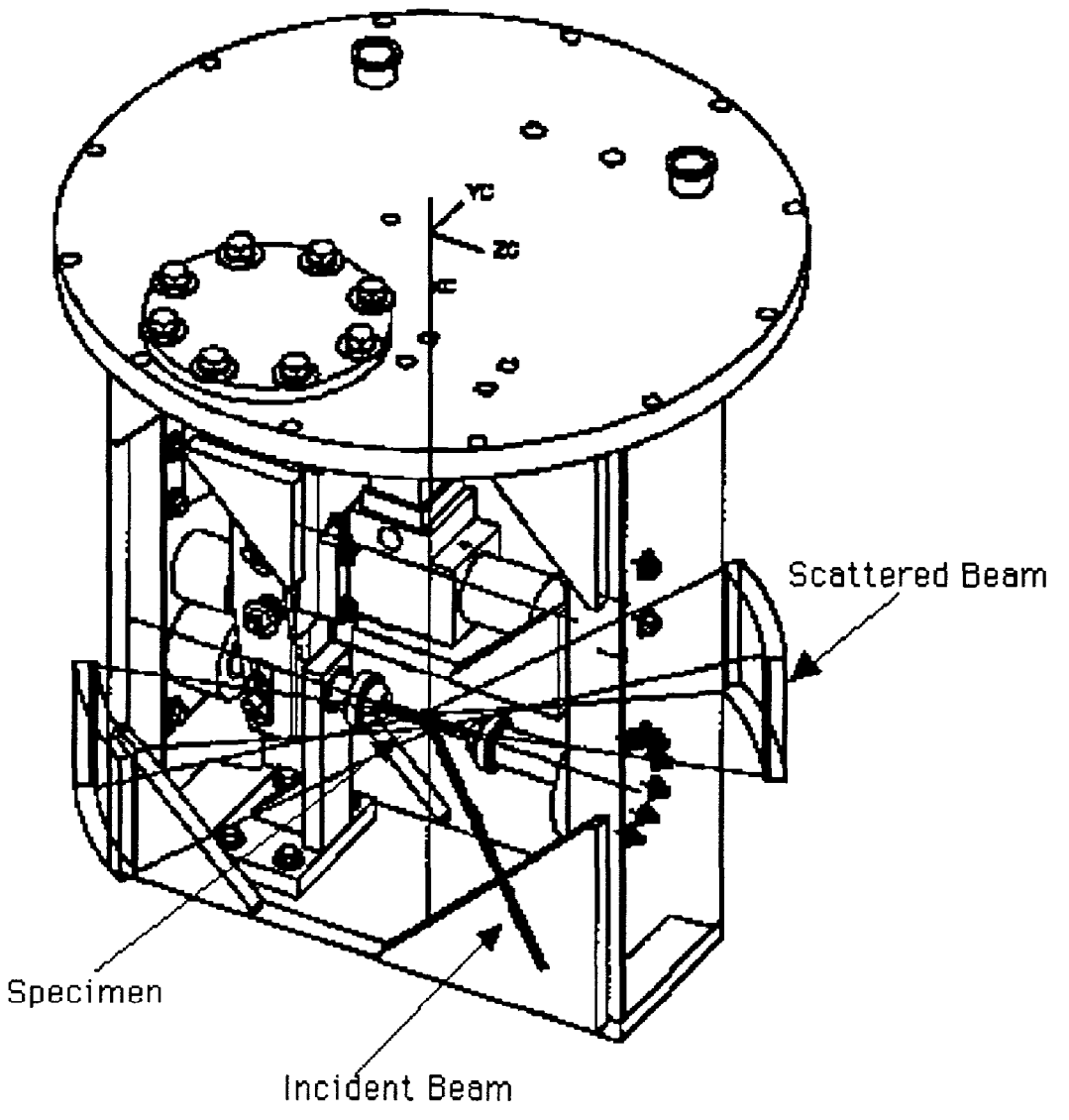

Figure 1: To operate a stress rig in the limited volume of the NPD sample chamber careful design was needed to ensure that the incident and diffracted beams were unimpeded.

Experiments:

Ford Motor Company in the US has been investigating a variety of metal matrix composite

systems for use in the automotive industry [12]. 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. In the summer of 1992, *in-situ* loading up to and beyond yield were made on two composite materials Al/TiC and Al/SiC. Only the results from the Al/TiC will be reported here. The Al/TiC was an artificially aged Al-2219 alloy reinforced with 15vol% 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 monotonic or cyclic loading.

A cylindrical specimen shape with a length of 160 mm and a diameter of 10 mm was used. The central portion of the specimen was in the neutron beam giving a gauge length of 14 mm and a total irradiated volume of 1100 mm^3. The rig was operated in cross-head displacement control and measurements were made at series of static tensile loads. The time for each measurement was approximately 4 hours at a beam current of 75 μA. Experiments were carried out under ambient temperature conditions. The initial loading sequence involved measurements at the static loads up to 200 MPa, in 50 MPa steps which maintained the samples in the elastic region. This was followed by unloading in one step, reloading to 200 MPa in one step, and finally 50 MPa step loads to 327 MPa for Al/TiC and 411 MPa for Al/SiC, which were sufficient to introduce 1% total strain as measured by a strain gauge. During final unloading, data were collected at 200, 100 and 0 MPa. Upon completion of the experiment, the Al/TiC was left with a total plastic strain of »0.67% and the Al/SiC with »0.61%.

Data were analyzed by two methods. To obtain a value of the lattice phase strain averaged over all reflections, the entire diffraction pattern was fit using the Rietveld refinement code GSAS[13]. To obtain information about the anisotropy of strain, peaks were fit individually using the same profile function as the Rietveld refinement. Figure 2 shows the (111) and (200) strains for the matrix. Figures 3 and 4 show the lattice parameter data obtained from the Rietveld refinements for the matrix and reinforcement with the numerical predictions from the finite element model. Strain is obtained from the initial unloaded value of the lattice parameters for the Al and reinforcements. See references 14 and 15 for further details.

Discussion:

Some interesting features can be discerned in figures 2-4. Polycrystalline anistropy in the Al was noted both parallel and perpendicular to the loading direction. In the parallel direction, it becomes apparent above 200 MPa (see figure 2) and results in the variation of final residual strains upon unloading. Perpendicular to the loading direction, there is a significant difference in the behavior of the Al [111] and Al [200] directions. Compared to the starting state, the latter is left in a state of »500μstrain tension compared to the initial matrix and the former is in »150μstrain compression. A similar effect was noted in the Al/SiC specimen. The origin of the difference has not been identified, but measurements are currently being made to assess whether any unusual texture is present and whether the effect is associated with Al or the composite.

In figures 3 and 4, the average measured phase strain (calculated from the lattice parameters) is displayed with the results of a finite element model. At this time, finite element modeling has

only be carried out for the Al/TiC composites. The composite was assumed to be infinite with cylindrical particles periodically embedded in the matrix. Two models were computed: one with no thermal residual strains (TRS) from cooling and one with residual strains developed from a ÎT of 180º C from 200º C [16]. The model without TRS does not give good agreement with the data, so only the model including TRS is shown with the data. In the direction parallel to loading, monotonic increases in the slope are observed. As the matrix strain rate decreases with increasing load, the particle strain increases. This indicates load transfer to the reinforcement as the matrix starts to yield plastically.

One interesting feature of the average matrix strain perpendicular to the loading direction is its "zigzag" behavior (see fig 5). Previous observation of this behavior in an Al/SiC composite has been explained by diffusional stress relaxation processes [2]. Our current data do not show a non-monotonic increase in the parallel direction so that diffusional relaxation cannot explain our observations. In addition, the stress decrease during a measurement was less than 1%, again indicating that relaxation does not play a signifcant role. We find that inclusion of TRS significantly changes the morphology of the strain behavior. Inclusion of TRS in the finite element model changes the region around the reinforcement which first plastically yields. Without TRS the plastic yield begins above the particle, parallel to the load direction. With the inclusion of TRS, plastic yield begins in the region perpendicular to the load direction. The Eshelby-type Mean Field Theory employed in explaining previous Al/SiC data [2,10] in not capable of predicting spatial fluctuations in the local strain fields. Further details are discussed in ref. 16.

Summary:

Despite the constrained volume of the NPD, we have constructed a compact stress rig for *in-situ* measurements. The ability of pulsed neutron sources to examine multiple phases and all lattice reflections simultaneously has been shown to be a valuable tool. Despite the assumption of a continuum for finite element modeling, comparison with the data have shown reasonable accurate predictions and insight into deformation mechanisms. In the future, we will make measurements above room temperature »300º C and measurements at total applied strains greater than 1%.

This work performed under the auspices of DOE contract W-7405-ENG-36.

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. Needlemann, "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. Needlemann, "Thermally and Mechanically Induced Residual Strains

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 Acd. 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 ICRS2, (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 Riso Int. Symp. on Mat. Sci., 489-496.

12. John. E. Allison and Gerald. S. Cole, "Metal Matrix Composites in the Automotive Industry: Opportunities and Challenges," Journal of Metals **45** (1993) 19.

13. Allen C. Larson and Robert B. Von Dreele, "GSAS - Generalized Structure Analysis System," Los Alamos National Laboratory, Unclassified Report -- LAUR-86-748.

14. M. A. M. Bourke, J. A. Goldstone, M. G. Stout, A. C. Lawson, and J. E. Allison, "Strain Measurement in Individual Phases of an Al/TiC Composite during Mechanical Loading," in Residual Stresses is Composites: Measurement, Modeling, and ThermoMechanical Modeling (ed. E. V. Barrera and I. Dutta, TMS , Proceedings 122nd TMS Annual Meeting) Denver CO, Feb 21-25, 1993.

15. M. A. M Bourke, J. E.. Allison, J. A. Goldstone, N. Shi, M. G. Stout, A. C. Lawson, "Measurement of the Strain in Individual Phases of Aluminum Titanium Carbide during Loading and Comparison with Prediction," in preparation for Scripta Met. Mater.

16. N. Shi, M. A. M. Bourke, J. A. Goldstone, M. G. Stout, A. C. Lawson, L. C. Davis, J. E. Allison, "Influence of Thermal Residual Stresses on the Development of the Average Lattice Elastic Strain during External Loading in the Al/TiC Composites, in preparation for Acta Metall Mater.

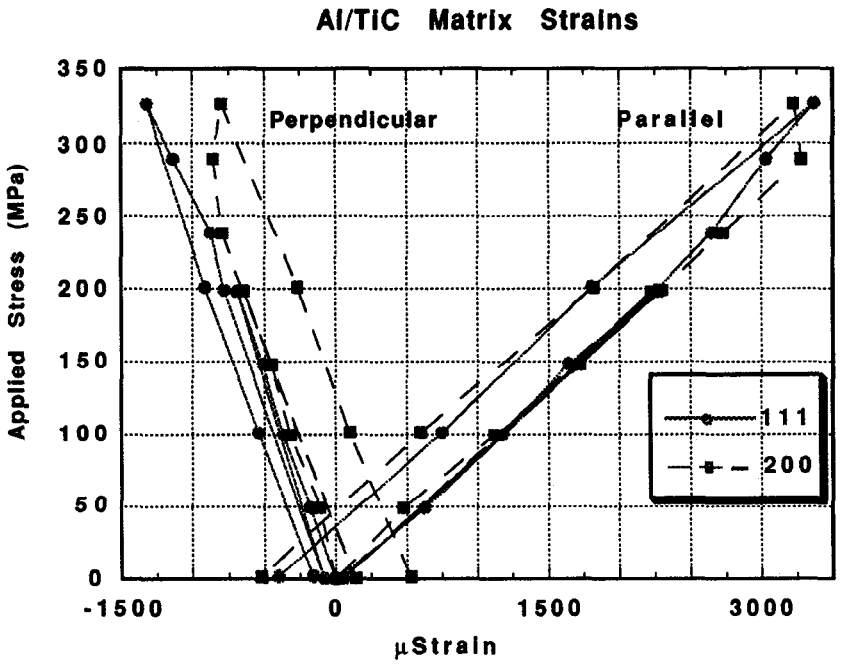

Figure 2: Strain data for the (111) and (200) reflections for the aluminum matrix of Al/TiC. Note the large difference in the (111) and (200) final residual strains in the direction perpendicular to the applied load. [ref. 14].

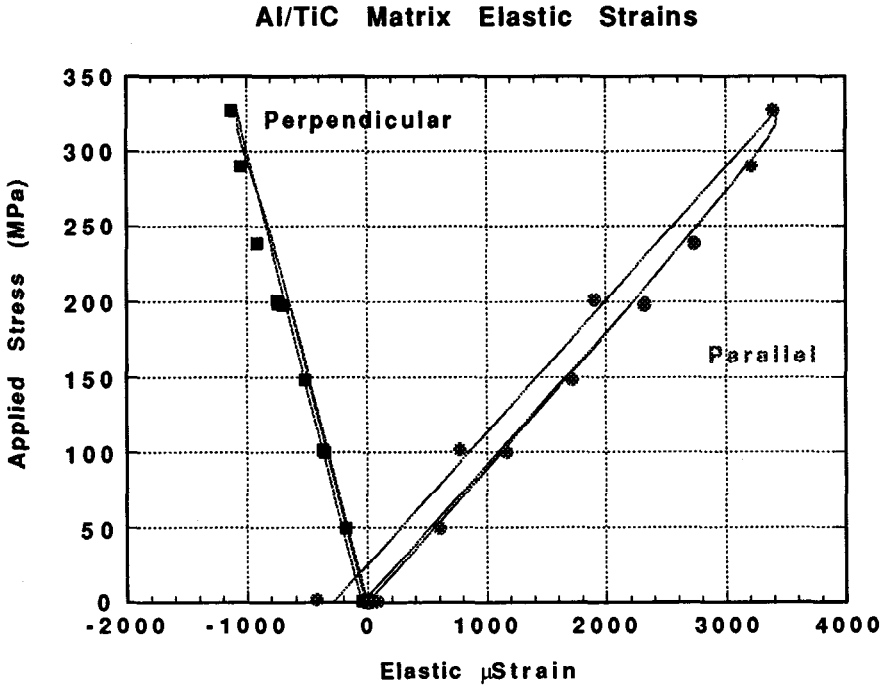

Figure 3: Elastic strain data (points) and FEM calculation [solid lines] for the average matrix strain in Al/TiC

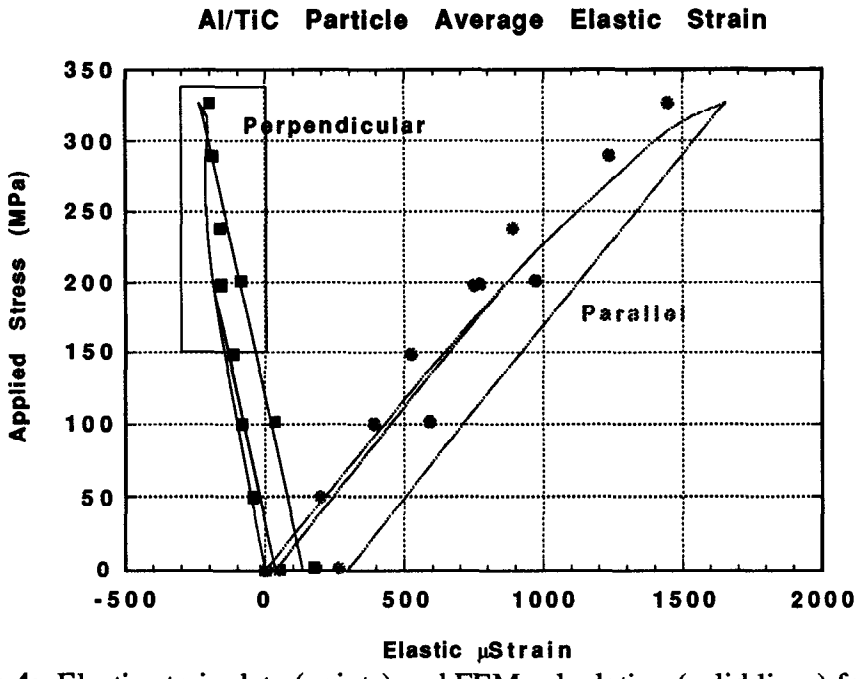

Figure 4: Elastic strain data (points) and FEM calculation (solid lines) for the average particle strain in Al/TiC. Boxed area is expanded in fig. 5.

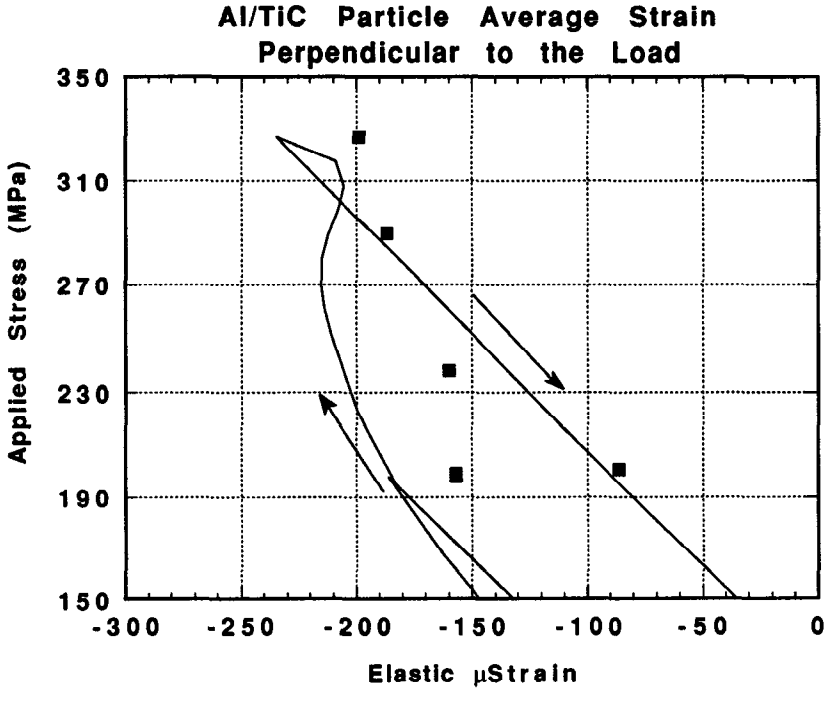

Figure 5: Particle elastic strain in the region of "zigzag" behavior (see zoom box in fig. 4).