LA-UR-96- 1033

Title: **Strain Measurement in Individual Phases of Multiphase Materials During Thermo-Mechanical Loading**

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Submitted to: DOE Office of Scientific and Technical Information (OSTI)

Los Alamos
NATIONAL LABORATORY

Strain Measurement in Individual Phases of Multiphase Materials During Thermo-Mechanical Loading

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Abstract

This is the final report for a three-year, Laboratory-Directed Research and Development (LDRD) project at the Los Alamos National Laboratory (LANL). Employment of metal matrix and ceramic composites in high-technology aerospace applications or as lighter (more economic) material in the auto industry requires the development of analytical methods capable of predicting the durability, debonding, and damage tolerance during the mechanical and thermal loads expected during service. Neutron diffraction has been used to measure residual stress in composites, steels, and compacted powders. We have extended our capability by acquiring a stress rig with a furnace to make in-situ measurements of materials response. This permits measurements on technologically important materials under conditions close to service. Studies address Al/TiC and Al/SiC composites (under consideration for automotive use) and $MoSi_2$.

1. Background and Research Objectives

Residual strains affect mechanical properties such as strength and fracture toughness, as well as the probability and origin of failure during use. As the demands of reliability and economy become more stringent the ability of designers to predict, understand and control the introduction of residual strains becomes more important. However the situation is complex because several physical processes may interact, including microstructural modification, phase transformations and plasticity. Numerical codes are commonly used to predict the development of such strains and the failure probability. However the complexity of the situation makes it imperative that predictions are validated by experiment during and after temperature and load conditions that simulate service.

The significance of this research cannot be overstated. Experience has shown that by controlling the microstructures of multiphase alloys, significant advances have been made in

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the properties of engineered materials. The measurement of internal strains and their evolution is of utmost importance in engineered alloy design because it provides information about the stress state of the material in the as-fabricated state and its ability to distribute the applied load between its constituents in service. Thus, it is not surprising that diffraction techniques that nondestructively measure residual strains in bulk components are being developed at most neutron sources. We have gone one step further developing a facility to measure strains at service temperature and under load. This has given us a unique capability and strong attraction for work on composite materials of interest to the transportation industries.

Our objectives were twofold. First, to improve our understanding of elastic and plastic deformation at a microstructural level; and second, to develop codes capable of predicting the strains during and after loading.

2. Importance to LANL's Science and Technology Base and National R&D Needs

In industrial manufacturing, residual strains introduced during fabrication result in a considerable waste of time and resources. Unfavorable residual stresses lead to distortion, cold-cracking and premature failure, necessitating rejection or repair with obvious economic consequences. The importance of residual stress analysis has been recognized as is apparent from its inclusion in a variety of LANL initiatives including heat treatment, shot peening, and machining. However the deformation of materials at a microstructural level is not clearly understood (even for single phase materials) because of directional anisotropy of randomly aligned grains in a polycrystal. As material systems become more complicated the need to understand deformation at a microstructural level increases. Before it is possible to predict the residual stresses we must understand the manner in which polycrystalline multiphase materials deform during applied loading. The capability to measure microstructural material response under load, in conjunction with subsequent residual stress measurement, significantly broadens LANL's technology base for partnership with industry. The ability of LANL to make "in-house" measurements is an advantage only available at a handful of other neutron sources in the US. When both load and temperature (1000° C) measurements are possible the capability is unique to LANL. This project supports a Los Alamos core competency in nuclear and advanced materials.

3. Scientific Approach and Results to Date

Stresses are not observed directly but are measured through their effects, strains. Strain is the change in length divided by the original length of the component. Conventional techniques for measuring residual strains suffer from drawbacks. Two of the most widely used are strain gauge sectioning and x-ray diffraction. Strain gauge measurements provide a bulk measurement but fail to distinguish between phases and are totally or partially destructive. X-rays are limited to surfaces that may not be representative of the interior of a component. By contrast, neutron diffraction offers nondestructive measurement of the strain in distinct phases due to applied or residual stress. Neutron diffraction is similar to x-ray diffraction, however neutrons provide information 10,000 times deeper in iron, for example, than x-rays. Neutron diffraction can validate conventional methods or computer predictions and may be used for complicated components for which conventional methods are impeded. No other nondestructive technique gives results from bulk materials which can be so directly compared to numerical predictions or can measure strain changes in individual phases.

At spallation sources, pulses of neutrons are used (instead of a continuous single wavelength). The wavelength of a detected neutron is determined from its time of flight between its creation by the spallation process and its arrival at the detector. Thus the specimen is scanned 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 approached more effectively using pulsed rather than monochromatic neutrons.

We have developed a compact stress rig for *in-situ* strain measurements on the neutron powder diffractometer (NPD) at the Los Alamos Neutron Scattering Center (LANSCE). The rig provides uniaxial compression and tension to 1000 MPa and is capable of heating a specimen to 1000° C in vacuum. The feasibility of in-situ stress measurement has been demonstrated on SiC reinforced aluminum composites and other composites.

Two materials, Al/SiC and Al/TiC, were initially studied at ambient temperature. Extensive analysis of the data from each experiment has been performed and published. Some of the results from the Al/TiC are described here.

The Al/TiC specimen was an artificially aged Al-2219 alloy (in T6 condition) reinforced with 15% vol. TiC. This system is very convenient for numerical modeling comparisons. The specimen was 160 mm long with a circular cross section and diameter at the gauge section of 10 mm. Only 14 mm in length at the center was illuminated by the neutron beam giving a sampling volume of approximately 1100 mm^3. An initial load cycle to 198 MPa was chosen to keep the specimen in the presumed elastic region (however, small modifications to the initial residual strains were noted on unload). Subsequent loading to 327 MPa induced 1% total strain and, on unload, a permanent plastic strain of approximately 0.67% as measured by the strain gauge. Figure 1 shows the measured strain for the most intense lattice reflections in each phase, parallel to the loading direction. The strains are calculated by noting the difference of the d-spacings from the unloaded spacings on the same specimen. Hence they do not include the initial thermal residual strains. Absolute strains for the TiC were determined by measuring a powder of the reinforcing particles dissolved from the matrix by acid. Parallel to the loading direction, there is only slight anisotropic yield for different aluminum reflections that results in a spread of the residual strains upon unload of less than 200 μstrain. Perpendicular to the loading direction, a more significant difference between the Al (200) and the Al (111) reflections was noted with considerable range in residual strains exhibited for different lattice directions. Thus, the material behavior in the aluminum perpendicular to the loading direction varies with crystalline orientation, and some grain scan exhibit strain that differs considerably from the bulk average.

A detailed analysis of this experiment using the finite-element code ABAQUSTM was carried out. 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 ΔT of 180° C from 200° C. The model without TRS does not give good agreement with the data, so only the model including TRS is shown with the data (Figure 2). 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 (Figure 2b). Previous observation of this behavior in an Al/SiC composite has been explained by diffusional stress relaxation processes. Our current data show a monotonic increase in the parallel direction so diffusional relaxation is not a valid explanation. In addition, the stress decrease during a measurement was less than 1%, again indicating that relaxation does not play a significant role. We find that inclusion of TRS significantly changes the morphology of the strain behavior. 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 is not capable of predicting spatial fluctuations in the local strain fields.

Measurements at potential service temperatures (110° - 150° C) on Al/TiC and Al/SiC complemented the work at ambient temperatures. The results are being examined in the context of relaxation and plasticity mechanisms. Preliminary interpretations indicate that the creep behavior of these materials under constant load can be interpreted using the same model developed to describe ambient temperature loading. This model has already helped in understanding deformation behavior by identifying the site of initial plasticity around reinforcement particles. Further modeling using ABAQUS is underway. A second composite system of interest to the aerospace industry, Al/Al_3Ti, in which both constituents can plasticity deform, exhibits a marked change in its deformation properties above 400°C. Measurements were made at room temperature and approximately 400° C to study the difference in stress partition due to elevated temperatures.

In collaboration with D. Dunand and D. Mari of Massachusetts Institute of Technology (MIT), we examined a very different metal matrix composite consisting of NiTi and TiC, conducting the first in-situ loading experiments on such materials. NiTi is a shape memory alloy that deforms by twinning from the high-temperature cubic phase (austenite) to the ordered low temperature monoclinic phase (martensite). As a result of self-accommodation during the transformation, which minimizes the internal strains, stress-free martensite consists of equal proportions of the 24 crystallographic variants. Upon deformation, the variant with the largest strain in the direction of the applied load grows at the expense of the other variants. This twinning deformation results in a preferred orientation in the martensite and a macroscopic strain. This strain can be recovered by heating the structure into the austenite region. Neutron diffraction measurements of the internal elastic strains and texture were performed during compressive loading of NiTi and a NiTi/TiC composite. Experimental conditions were similar to those described above but in this case we loaded in compression and the samples were cylinders 10 mm in diameter and 24 mm long. Diffraction measurements were recorded at uniaxial compressive stresses of -3, -90, -210, and -280 MPa, to a maximum macroscopic strain of 1.6%. Further measurements were made during unloading at stresses of -100 and -3 MPa. The samples were then recovered by heating above the austenite transformation temperature and measured a final time.

Upon loading, the intensity of the (100) planes perpendicular to the load axis increases while that of the (011) planes decreases. The opposite behavior is observed for the planes parallel to the load axis. We concluded that variant coalescence produces preferential orientation of the (100) planes perpendicular to the compressive load axis. The degree of preferred orientation is directly related to the amount of plastic strain for the (100) and (001) planes as shown in Figure 3. To the best of our knowledge this is the first time this approach has been applied to martensite twinning. Upon unloading, the stress-strain curve did not appear linear, indicative of partial recovery during unloading. This represents the first direct observation in the bulk of the reversibility of the twinning upon mechanical unloading and shape-memory recovery. The final observation of this study was the lack of any modification of the results due to the addition of the TiC particles.

Using the new stress rig capability to heat to 1000° C we measure the elastic properties of steel phases at different temperatures. By performing diffraction strain measurements under known loads and at varying temperatures the respective mechanical properties of austenitic and martensitic phases can be measured. At temperatures between ambient and 800°C, simultaneous determinations of the phase volume fractions and their elastic properties are made. The results are benchmarked against other experimental approaches including the ultrasonic resonance technique. The principal material studied is a quenched SAE 5180 steel because of its importance to an ongoing CRADA – Predictive Model and Methodology for Heat Treat and Distortion. The goal is to develop a generally applicable finite-element analysis package, with constitutive relationships appropriate to specific materials, that can predict strains and distortions for a variety of heat treatments and component geometries. Experimental determinations of the temperature dependence of the individual phase mechanical properties provide a valuable test of the constitutive relationships included in the models.

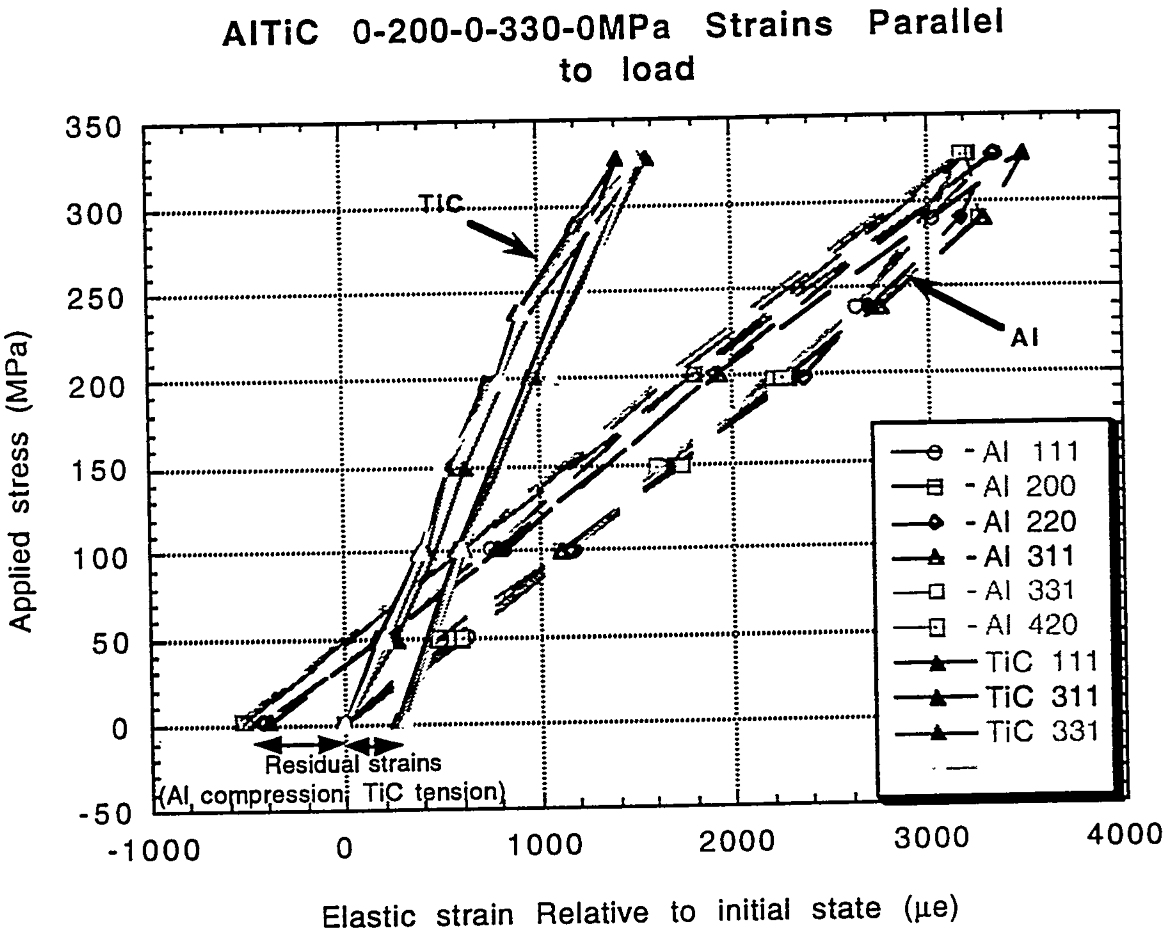

Figure 1: Elastic strain (relative to the unloaded state) measured by neutron diffraction for an Al/TiC composite in the direction parallel to the load for several Al and TiC reflections.

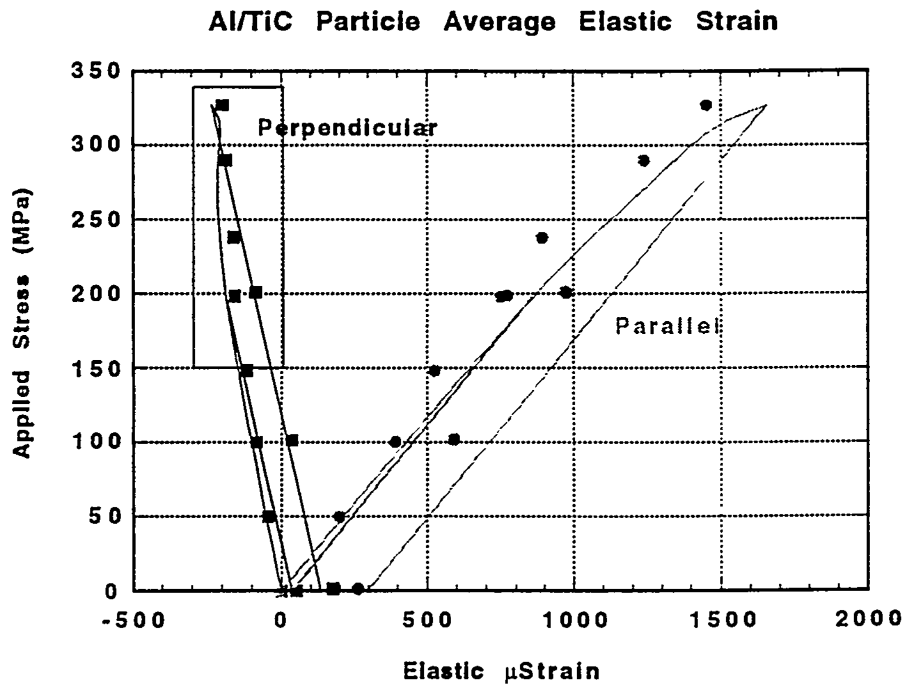

Figure 2a: Elastic strain data (points) and finite element model calculation (solid lines) for the average particle strain in Al/TiC. Boxed area is expanded in fig. 2b.

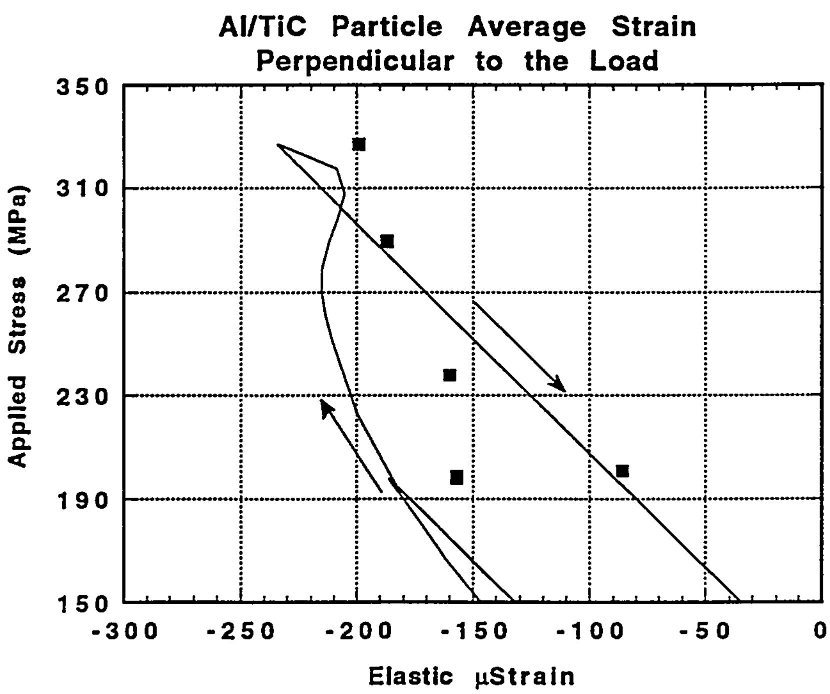

Figure 2b: Particle elastic strain in the region of "zigzag" behavior (see zoom box in fig. 2a).

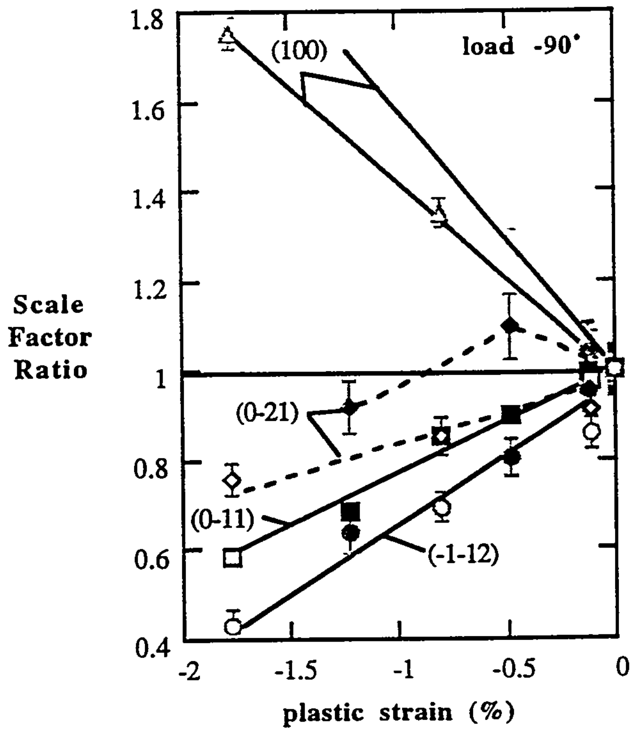

Figure 3: Normalized scale factor upon mechanical loading as a function of plastic strain for NiTi (filled symbols) and NiTi-20% TiC (empty symbols) for planes perpendicular to the applied stress.