Conf-941144--11

LA-UR- 94-4043

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Submitted to: Materials Research Society, Symposium BB, Neutron Scattering in Materials Science, Boston, MA, Nov 28-Dec 2, 1994

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Form No. 836 R5
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INTERNAL STRAIN MEASUREMENT USING PULSED NEUTRON DIFFRACTION 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 in the only technique that can make nondestructive measurements in the interior of components. By recording the change in crystalline lattice spacings, elastic strains can be measured for individual lattice reflections. Using a pulsed neutron source, all lattice reflections are recorded in each measurement, which allows for easy examination of heterogeneous materials such as metal matrix composites. Measurements made at the Manuel Lujan Jr. Neutron Scattering Center (LANSCE) demonstrate the potential at pulsed sources for in-situ stress measurements at ambient and elevated temperatures.

INTRODUCTION

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. 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. Thus residual stress between phases is common. The situation is more complex because in addition to crystalline anisotropies[1], physical processes like fiber breakage and plastic or diffusional relaxation may interact during production and service. Numerical codes[2-4] are frequently used to predict the development of internal strains as a result of such processes: the complexity of the situation makes experimental validation important during and after thermomechanical conditions that simulate service. Neutron diffraction provides a unique method for examining materials during thermomechanical loading because it is nondestructive and penetrating and can distinguish between the strains in individual phases. 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 thermomechanical loading. Neutron diffraction offers nondestructive measurement of the average phase strain from volumes of a few mm^3 up to several cm^3.

NEUTRON DIFFRACTION FUNDAMENTALS

Diffraction methods of measuring strain by x-rays[5,6] or neutrons[7-10] have been extensively covered in the literature and only a brief overview is given here. Changes in

the lattice spacings of crystalline materials experiencing a residual or applied stress are the basis of strain measurement by diffraction. A pulsed source operates in what is called time of flight (TOF) mode. Discrete pulses of neutrons are produced by a process called spallation which occurs when energetic protons interact with a heavy metal target. The neutrons in each pulse constitute a continuous energy spectrum with a distribution determined by the characteristics of the 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 using Bragg's Law. 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. 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:

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

where t_{hkl} is the time of flight for a particular *hkl* reflection. 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. 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, thus pulsed sources are superior.

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 2×10^{-3}. Particularly for the ceramic reinforcement strains are often less than 10^{-3} and high-resolution instruments are needed to discern them.

CURRENT LANSCE CAPABILITIES

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 four directions by four detector banks at ±90° and ±148°, measured from the incident beam. The best resolution is achieved in the backscattering banks, ±148°. In practice each detector bank subtends an angle typically about 10° corresponding to a range in Q of 5°. The strains measured are along the directions bisecting the incident and diffracted beams.

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

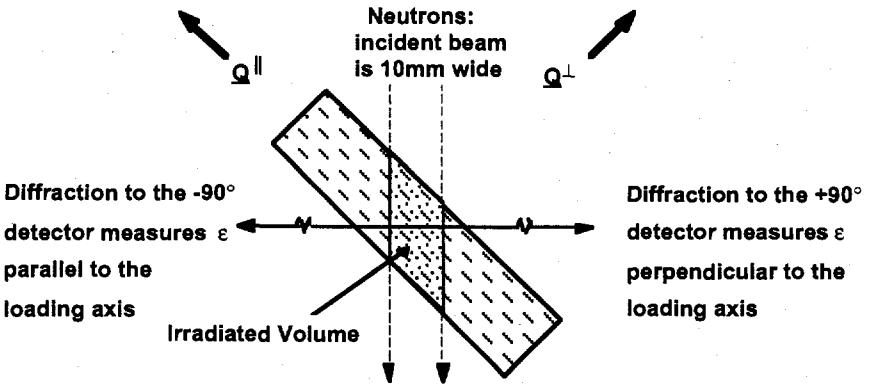

Figure 1 Schematic of specimen orientation in the NPD for a uniaxial load experiment (the backscattering detectors are not shown).

actuator was in parallel with the specimen and the load was transferred to the specimen through a pivot arm[13]. 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 (see figure 1) and at 61° in one backscattering bank. For a typical specimen diameter of 10 mm, the irradiated volume is 1000 mm^3. The load is applied in increments and elastic strains are obtained under either constant load or constant displacement. The maximum load is approximately 50 kN (or about 1 GPa on a 6 mm diameter sample). Grips for both compression and tension are available. Halogen lamp heaters are currently installed allowing measurements up to sample temperatures of 1000° C.

EXPERIMENTS ON Al/SiC COMPOSITES

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 nonlinearity in plots of applied loading vs. elastic strain in individual phases of a composite is indicative of load transfer. Although nonlinearity does not provide unambiguous identification of the active mechanisms it does offer a test for material models[1]. 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.

Many of the automobile applications of Al/SiC such as connecting rods, brake rotors or drive shafts are expected to see sustained or periodic temperature fluctuations. In 1992-3 we performed static uniaxial tensile experiments on samples made of Al/SiC and Al/TiC at ambient temperatures. The results for these earlier experiments have been compared to finite element (FE) modeling that has already provided insight into the deformation and site of initial plasticity in particle reinforced composites[13]. On examination of the effective strain contours in the FE model it was shown that the

presence of the thermal residual stress alters the strain field so that the site of matrix initial yielding changes which in turn alters the morphology of the loading curve.

In this example we present strains measured in a uniaxial tension test on a 15 vol% SiC Al material (supplied by DWA). The samples were 160 mm long with a circular cross section and a diameter at the gauge section of 10 mm. One inch long (125W) cartridge heaters at either end of the sample controlled the center at 110°C. The specimen was surrounded by a vanadium heat shield. The strains parallel to the loading direction are shown in figure 2. For clarity only a few reflections are given. The inset shows the macroscopic strain recorded using a strain gauge. Each measurement at a static load level took approximately 4 hours. Above 200 MPa 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\varepsilon$) relative to the starting state of the material and the silicon carbide in compression (< -100$\mu\varepsilon$). Different reflections showed different residual strains with the largest occurring for the Al 200 which was close to 500$\mu\varepsilon$, evidence for which can be seen in figure 2 where it bends away from the three other aluminum reflections. Providing that the morphology of the loading curves can be identified with sufficient accuracy inferences about the material deformation can be made[12].

To examine the creep behavior of the reinforcement, again a uniaxial tension specimen was used. In this experiment successive loads of 100 MPa, 200 MPa, 300 MPa, 365 MPa, 400 MPa, 410 MPa, and 425 MPa were applied to the sample (see fig. 2). Data at each load level above 400 MPa were collected in 60 minute intervals for 4 intervals. While full analysis of these data are not yet complete, the behavior under constant load conditions is predicted by the same model that explains the ambient temperature loading data. Partial plasticity at of the Al matrix occurs at 400 MPa as evidenced from the reduction in elastic strain over the duration of the measurements, while at 425 MPa full plasticity of the matrix is verified by the absence of any strain reduction.

The strength of the neutron diffraction technique lies in its ability to separate average phase strains in the bulk, 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. 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.

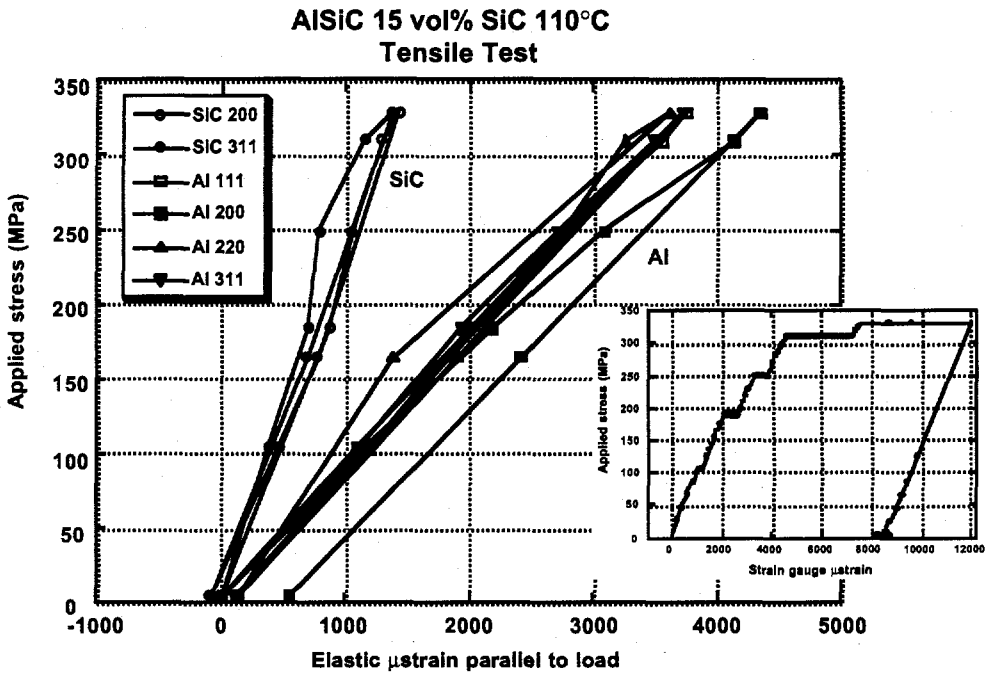

Figure 2: Parallel strain relative to initial material state in a uniaxial tension test of a 15 vol% SiC Al 6091 (T6) particulate MMC.

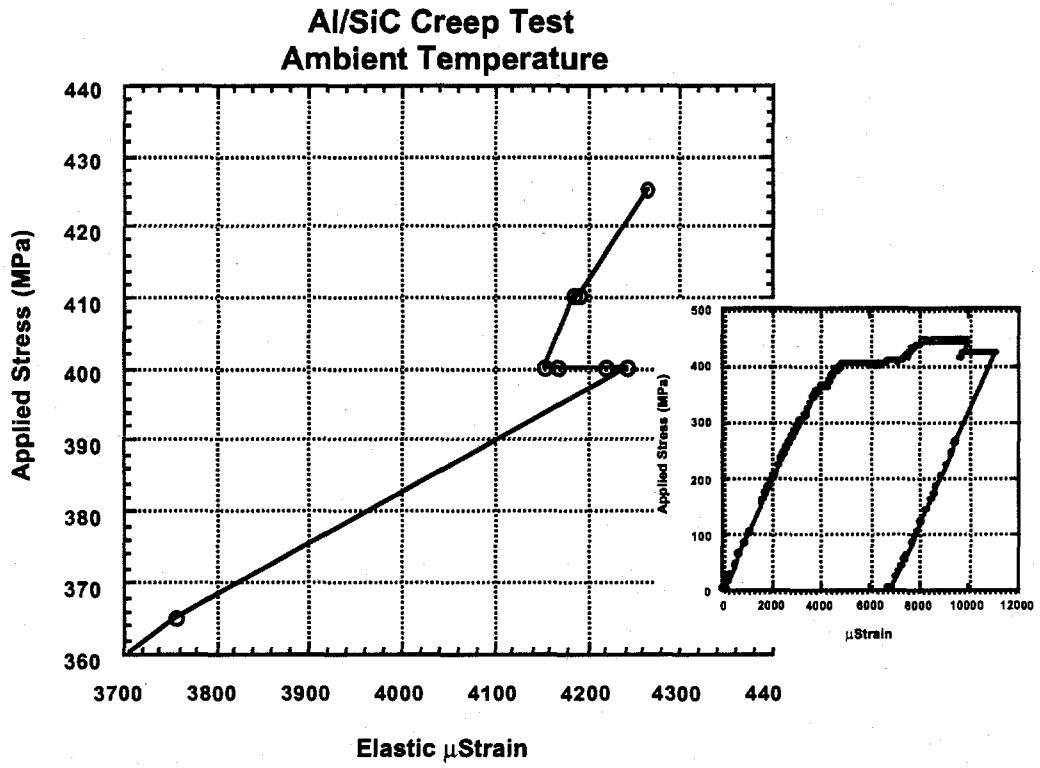

Figure 3: Parallel elastic composite macrostrain relative to initial material state in a uniaxial tension fracture test of a 15 vol% SiC Al particulate MMC. Data were taken in 60 minute intervals at a given load.

SUMMARY

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. 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. Equally there is the possibility of studying combinations of stress and/or temperature induced phase transformations in a variety of material systems. Finally, comparison of the experimentally determined neutron volume average with the FE cell volume average is an indirect tool for probing the localized strain behavior.

This work supported by the U. S. Department of Energy, contract W-7405-ENG-36.

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