Conf-920624--1

APPLICATION OF NEUTRON DIFFRACTION TO MEASURE RESIDUAL STRAINS IN HIGH TEMPERATURE COMPOSITES

A. Saigal
Department of Mechanical Engineering
Tufts University
Medford, MA 02155
(617)-381-3239

and

D.S. Kupperman
Materials and Component Technology Division
Argonne National Laboratory
Argonne, IL 60439
(708)-972-5108

ANL/CP--73280

DE91 014021

For Presentation and Publication in Conference Proceedings

Sixth Japan–U.S. Conference on Composite Materials
Orlando, Florida
June 22–25, 1992

APPLICATION OF NEUTRON DIFFRACTION TO MEASURE RESIDUAL STRAINS IN HIGH TEMPERATURE COMPOSITES

A. Saigal
Department of Mechanical Engineering
Tufts University
Medford, MA 02155

and

D. S. Kupperman
Materials and Component Technology Division
Argonne National Laboratory
Argonne, IL 60439

Abstract

An experimental neutron diffraction technique was used to measure residual thermal strains developed in high temperature composites during postfabrication cooling. Silicon carbide fiber–reinforced titanium aluminide (over the temperature range 20–950°C) and tungsten and saphikon fiber–reinforced nickel aluminide composites (at room temperature) were investigated. As a result of thermal expansion mismatch, compressive residual strains and stresses were generated in the silicon carbide fibers during cooldown. The axial residual strains were tensile in the matrix and were lower in nickel aluminide matrix as compared to those in titanium aluminide matrix. The average transverse residual strains in the matrix were compressive. Liquid–nitrogen dipping and thermal–cycling tend to reduce the fabrication–induced residual strains in silicon carbide fiber–reinforced titanium

aluminide matrix composite. However, matrix cracking can occur as a result of these processes.

Introduction

The mechanical performance of most engineering materials, including high temperature composites, is influenced by process–induced residual strains and stresses that are locked in the constituents during cooling after fabrication. Silicon carbide fiber reinforced titanium matrix composites are currently being evaluated for structural applications in jet engines because of their low weight, high strength and high stiffness potential at high temperatures. In addition, tungsten and saphikon fiber reinforced nickel aluminide matrix composites are also being investigated.

High–temperature composites are usually fabricated at temperatures above 900°C and as there is usually a significant mismatch between the coefficients of thermal expansion of the fibers and the matrix, the thermally induced residual strains and stresses can be significant. In many engineering composites, frictional forces at the interfaces often provide the necessary link between the reinforcements and the matrix, because chemical bonding is either weak or nonexistent (1,2). Since frictional forces and subsequent behavior of the composites depend on the residual stresses that develop during cooldown after fabrication, it is important to have an idea of the residual strains and stresses that exist in the composites.

For all composites with crystalline constituents, neutron diffraction is a powerful tool for measuring bulk elastic residual strains from which residual stresses can be calculated in much the same way as is done with X-ray diffraction (3, 4). Such stresses are called microstresses, because they vary on the scale of the microstructure. These are in sharp contrast to macrostresses that are typically measured with X-rays in the near-surface region, and that commonly arise from joining, e.g. welding, and surface finishing. Unlike macrostresses, which are amenable in principle to destructive as well as non-destructive methods, microstresses must be measured using diffraction. X-rays have been used to determine microstresses in systems with relatively light elements, such as Al/SiC composites (5). However, neutron diffraction offers significant advantages over X-rays, particularly when heavy elements, laminar microstructures or concurrent presence of micro- and macro-stresses are involved. Further, unlike X-rays, neutrons can penetrate deep into the interior of most engineering materials, typically a factor of 1000. This provides for an excellent volume sample, a truly bulk measurement of the residual strains, and independence from surface-related effects.

Allen et al. used a neutron diffraction method to measure residual stresses and load-induced bulk stresses in a metal matrix composite (6). Kupperman et al. used a neutron diffraction technique to study the variation of residual strains and stresses in a SiC whisker-reinforced Al_2O_3 matrix composite with temperature and volume fraction of whisker (7). The obtained results agreed

reasonably well with calculated values (8). Krawitz et al. measured residual stress, as a function of temperature, in a high volume fraction WC–Ni cemented carbide composite (9). In addition, Majumdar et al. have used the intense pulsed neutron source (IPNS) and the general purpose powder diffractometer at Argonne National Laboratory to measure residual strains in a number of engineering composite materials (10).

In the present study, the neutron diffraction technique was used to measure residual thermal strains, developed during cooling,in silicon carbide fiber–reinforced titanium aluminide and tungsten and saphikon fiber–reinforced nickel aluminide, high–temperature composites. The effects of fabrication procedures and thermal processing, such as liquid–nitrogen dipping and thermal–cycling, on the residual strains were also studied.

Neutron Diffraction Measurements

Thermal neutrons with wavelengths on the order of the lattice spacings are used in the experiments. Bragg's Law of diffraction can then be applied to neutrons as follows:

$$2\, d_{hkl}\, \sin\theta = \lambda_{hkl} \qquad (1)$$

where d_{hkl} is the lattice spacing, 2θ is the angle between the incident and the scattered neutron beams when a Bragg peak is detected, λ is the de Broglie wavelength of the neutron, and *hkl* are the Miller indices of the diffracting planes. The data were collected using the General Purpose Powder Diffractometer (GPPD) at the

Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory. The GPPD is a time-of-flight instrument. The spectrum is measured at a fixed Bragg angle 2θ of ± 90 degrees. The main advantage of a pulsed neutron source over a steady neutron source is that it uses a "white" spectrum of neutron energies. Therefore, during a single measurement at IPNS, many diffraction peaks, i.e., crystallographic directions, of each phase are recorded simultaneously in various spatial directions.

In applying neutron diffraction, the lattice spacings in various crystallographic directions of stress-free powders and/or fiber (which are used to fabricate the composite) are determined first. The shifts in the Bragg peaks of the stresses constituents of the composite are then determined. For any $\{hkl\}$ diffraction peak, the lattice strain is given by

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_0}{d_0} \qquad (2)$$

where d_{hkl} and d_0 represent the average interplanar spacings in the stressed and unstressed lattice, respectively. With a pulsed source, changes in lattice spacing are related to time-of-flight shifts in the Bragg peaks. The relationship of time-of-flight to neutron wavelength is described by the equation

$$\lambda = ht/mL \qquad (3)$$

where h is the Planck's constant, t is the time of flight for a neutron to reach a detector after leaving its source, L is the flight path of the

sample. Lattice parameters for various crystallographic directions were measured, parallel and perpendicular to the fiber axis. The diffraction peak that provided the most useful data for silicon carbide fibers was that for the {220} plane. For the matrix, the 1.75 Å line provides the average residual strains in the matrix. Figure 2 shows the measured average residual strains in the SCS–6 fibers and the Ti–14Al–21Nb matrix, parallel to fiber, as a function of temperature. At room temperature, the strain in the fiber is compressive (–0.0019) and the strain in the matrix is tensile (+0.0042). As expected, the residual strains decreased as the temperature was increased from room temperature to the processing temperature, approaching a value of zero and a strain–free state around 806°C. This indicates that during the early cool down period following fabrication at 950°C, the thermal stresses generated in the composite are possibly quickly relaxed out by creep. At room temperature, the transverse strains in the fiber and matrix are both compressive, –0.0005 and –0.0007 respectively, and similar to the axial strains, decrease as the temperature is increased from room temperature to the processing temperature.

The relative increases in the fiber and matrix lattice spacings with increasing temperature, relative to the room–temperature value, for directions parallel and perpendicular to the fiber axis are shown in Figures 3 and 4. The similarity in expansion curves (parallel to fiber) for various crystallographic planes of the matrix indicates a state of plane strain in the sample. In addition, the anisotropy of the matrix can be clearly seen in Figure 4, where the

expansion curves for different crystallographic directions vary significantly for the matrix. This is most likely the result of small thickness of the sample perpendicular to the fiber axis.

In order to reduce the residual strains and stresses, the fabrication procedures and the thermal history of the composites can be altered. Two commonly used procedures are liquid-nitrogen dipping (LND) and thermal cycling. Figure 5 compares the measured residual strains in as-fabricated, liquid nitrogen dipped, and thermally cycled (100 times to 650°C) specimens. It can be seen that compared to the as fabricated sample, tensile strains in the matrix and compressive strains in the fibers (parallel to fibers) are lower for the LND and thermally cycled specimens. Thermally cycling appears to reduce the residual strains more than liquid nitrogen dipping. It was also found that the matrix and fiber strains perpendicular to the fibers did not change significantly with processing.

Using Hooke's Law, the residual stresses in the fibers can be calculated from the residual strains. Based on Table 1, there is no doubt that the axial stresses (parallel to fiber) in the fibers, and to certain extent stresses perpendicular to fiber, are reduced by the processing methods discussed. The limited plastic deformation of the matrix is not sufficient to reduce the observed reductions in residual strains and stresses. It is possible that the matrix is cracked due to thermal processing. The possibility of matrix cracking is further suggested by elastic moduli measured by

ultrasonic velocity of sound propagating perpendicular to the fibers. Decreases in Young's modulus for liquid nitrogen dipped (134 GPa) and thermally cycled (131 GPa) samples were measured as compared to the as-fabricated (137 GPa) sample.

Table 2 shows the measured strains in the matrix in various composites. The axial residual strains are tensile in the matrix and are lower in nickel aluminide matrix as compared to those in titanium aluminide matrix. It also shows that the strains in the nickel aluminide matrix due to 35 v/o tungsten fibers and 30 v/o saphikon fibers are similar.

Conclusions

Residual thermal strains in a 35 volume percent silicon carbide (SCS-6) fiber reinforced titanium alloy (Ti-14Al-21Nb) matrix, high temperature composite were measured, as a function of temperature, by neutron diffraction. The residual strains (parallel to fiber) decreased as the temperature was increased from room temperature to the processing temperature, approaching a value of zero and a strain-free state around 806°C. This indicates that during the early cool down period following fabrication at 950°C, the thermal stresses generated in the composite are possibly quickly relaxed out by creep.

The similarity in expansion curves (parallel to fiber) for various crystallographic planes of the titanium aluminide matrix indicates a state of plane strain in the sample.

It has been shown that significant residual strains and stresses develop in these composites during the cooldown from the processing temperature to room temperature. The silicon carbide fibers are under compressive residual strains and stresses. The strains in the matrix (parallel to fiber) are tensile and are lower in nickel aluminide matrix as compared to those in titanium aluminide matrix. The average transverse strains in the matrix are compressive.

Liquid–nitrogen dipping and thermal–cycling tend to reduce the fabrication–induced residual strains in silicon carbide fiber–reinforced titanium aluminide matrix composite. However, matrix cracking can occur as a result of these processes.

Acknowledgements

This work was supported by the U.S. Department of Energy, under Argonne National Laboratory discretionary funds and by General Electric Co. This work benefitted by the use of the Intense Pulsed Source at Argonne National Laboratory. The authors wish to thank P. K. Wright for supplying the samples and R. L. Hitterman for assistance in data acquisition.

References

1. D.C. Phillips, "Interfacial Bonding and Toughness of Carbon Whisker Reinforced Glass and Glass–Ceramics," Journal of Materials Science, Vol. 9, No. 11, 1974, pp.1847–54.

2. K.M. Prewo and J.B. Brennan, "High–Strength Silicon Carbide Fiber–Reinforced Glass–Matrix Composites," Journal of Materials Science, Vol. 15, No. 2, 1980, pp. 463–68.

3. M.T. Hutchings and C.G. Windsor, "Industrial Applicatien of Neutron Scattering"; Ch. 25 in Neutron Scattering, Edited by K. Skold and D.L. Price, Academic Press, New York, 1986.

4. I.C. Noyan and J.B. Cohen, Residual Stress, Springer–Verlag, New York , 1987.

5. P. Predecki, A. Abuhasan, and C.S. Barrett, "Residual Stress Determination in Al_2O_3/SiC (Whisker) Composites," Advances in X–Ray Analysis, Vol. 31, 1988, p. 231.

6. A.J. Allen, M. Bourke, M.T. Hutchings, A.D. Krawitz, and C.G. Windsor, "Neutron Diffraction Measurements of Internal Stress in Bulk Materials:–Metal Matrix Composites," Residual Stresses in Science and Technology, E. Macherauch and V. Hauk, Eds., DGM Informations–gelsellschaft Verlag, Oberursel, Vol. 1, 1987, pp. 151–57.

7. D.S. Kupperman, S. Majumdar, S.R. MacEwen, R.L. Hitterman, J.P. Singh, R.A. Roberts, and J.L. Routbort, "Nondestructive Characterization of Ceramic Composite Whiskers with Neutron Diffraction and Ultrasonic Techniques," Review of Progress in Quantitative Nondestructive Evaluation, Vol. 7B, D.O. Thompson and D.E. Chimenti, Eds., Plenum Publishing Corporation, 1988, pp. 961–69.

8. S. Majumdar, D.S. Kupperman, and J.P. Singh, "Determination of Residual Thermal Stresses in a SiC–Al_2O_3 Composite Using Neutron Diffraction," Journal of American Ceramic Society, Vol. 71, No. 10, 9188, pp. 858–63.

9. A.D. Krawitz, D.G. Reichel, and R.L. Hitterman, "Residual Stress and Stress Distribution in a WC–Ni Composite," Journal of Materials Science and Engineering, Vol. A119, 1989, pp. 127–34.

10. S. Majumdar, J.P. Singh, D. S. Kupperman, and A.D. Krawitz, "Application of Neutron Diffraction to Measure Residual Strains in Various Engineering Composite Materials," Journal of Engineering Materials and Technology, Vol. 113, January 1991, pp. 51–59.

Table 1 Residual strain and stress in fibers in as-fabricated, liquid nitrogen dipped, and thermally cycled Ti alloy matrix/SiC fiber composites

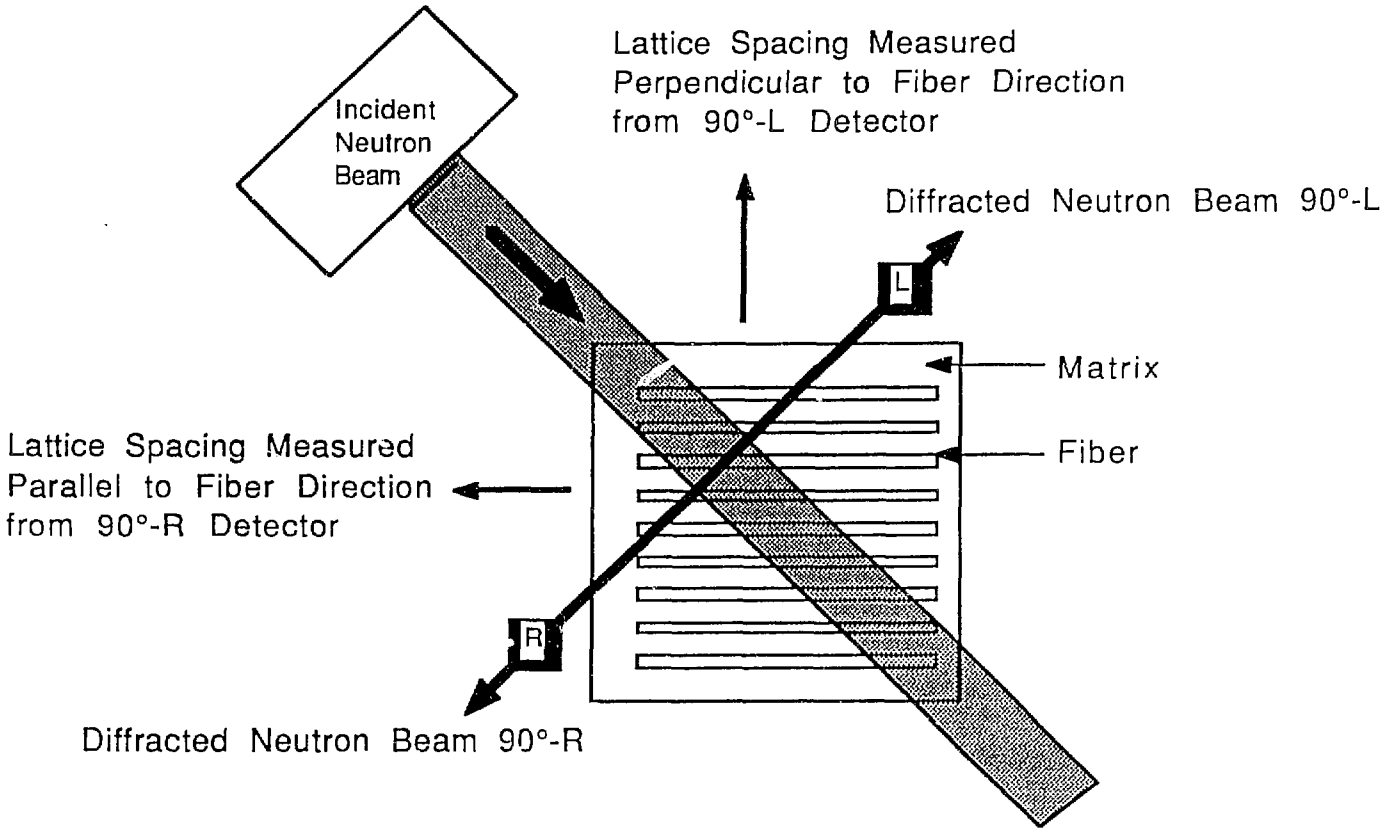
	Parallel to Fiber		Perpendicular to Fiber	
Processing	Strain	Stress (MPa)	Strain	Stress (MPa)
As-fabricated	-0.0019	-970	-0.0005	-483
Liquid Nitrogen Dipped	-0.0012	-652	-0.0005	-408
Thermally Cycled	-0.0008	-470	-0.0005	-366

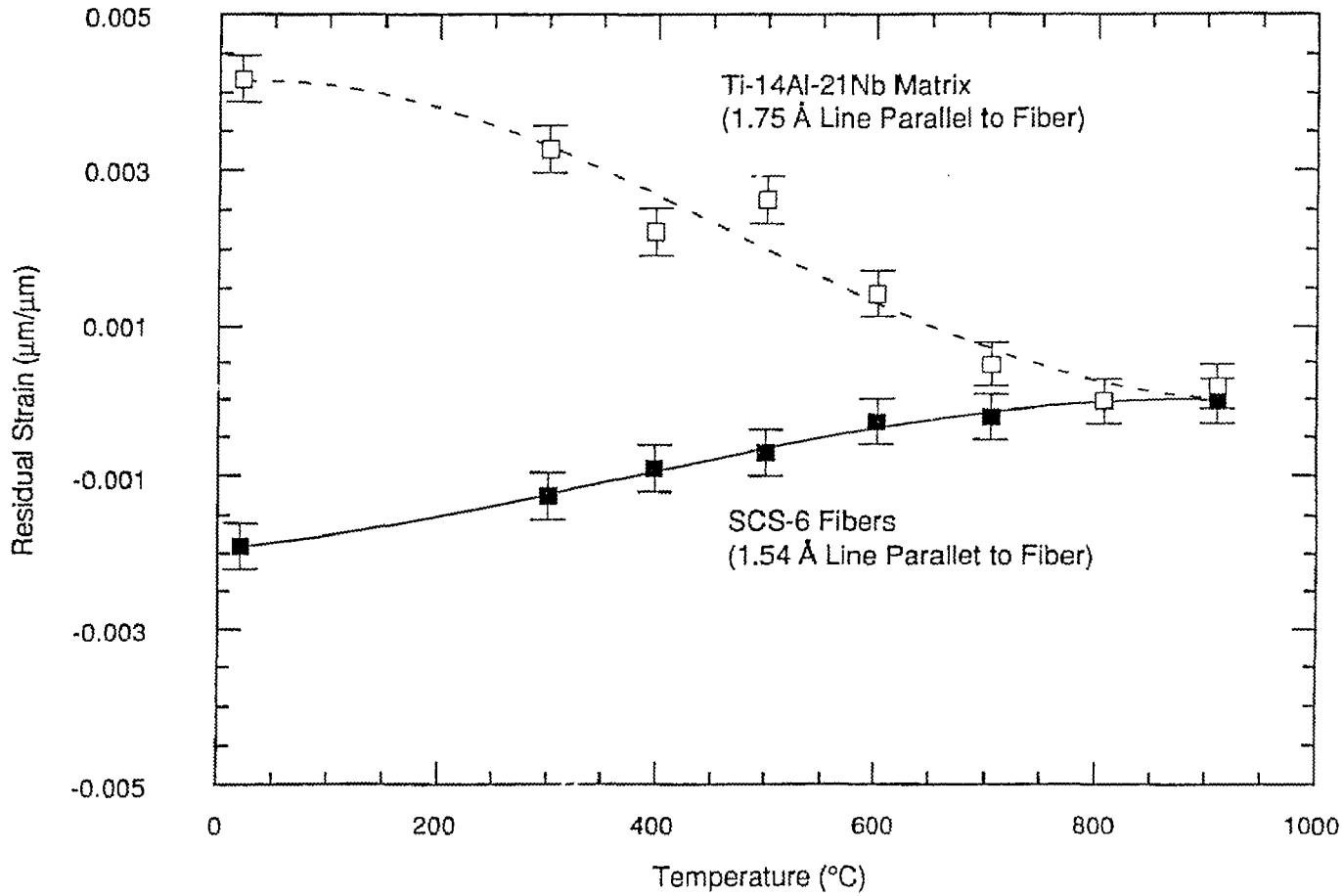
assuming E (fiber) = 414 GPa and ν (fiber) = 0.19

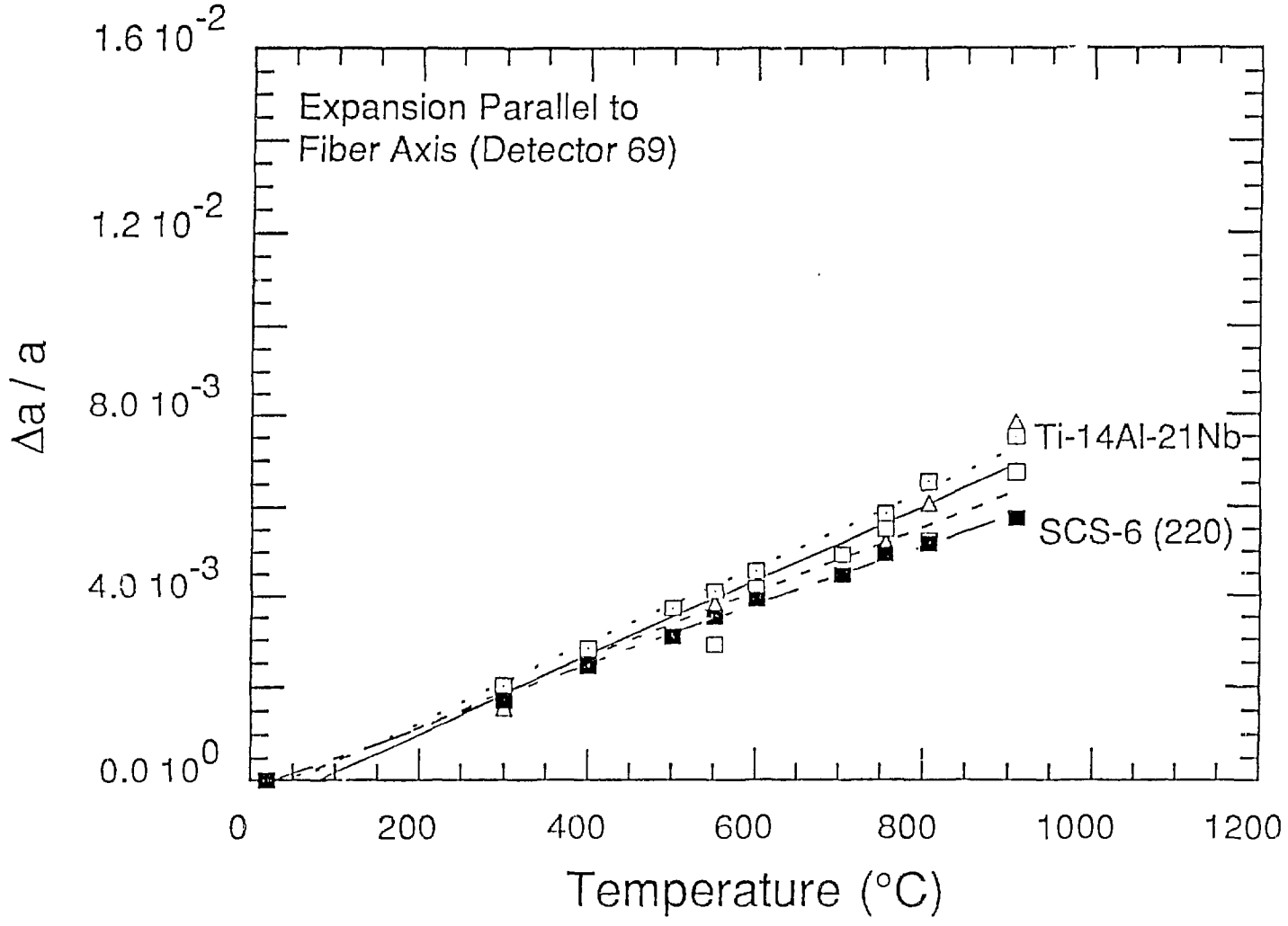
Table 2 Residual Strains in Matrix

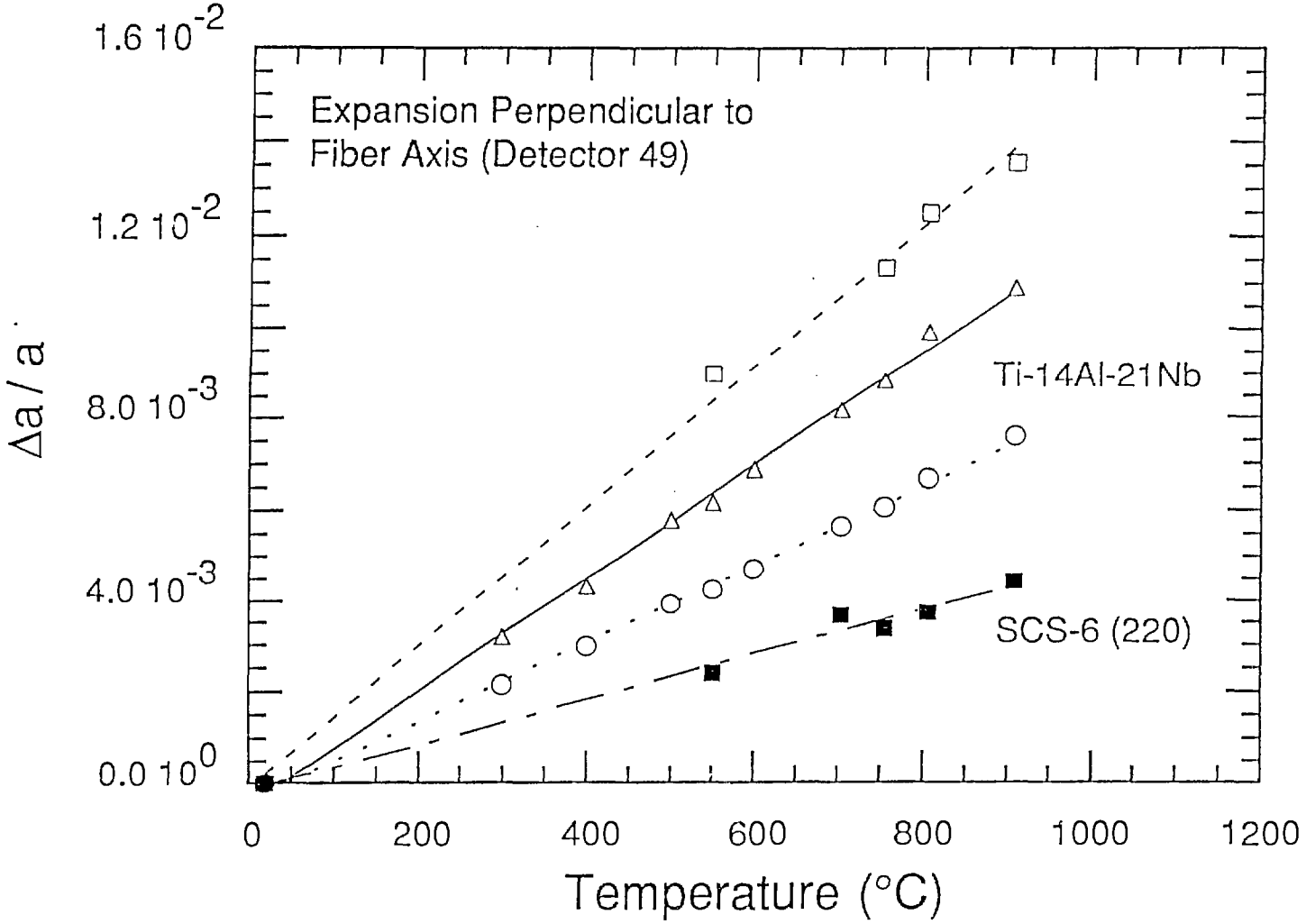
Matrix	Parallel to Fiber	Perpendicular to Fiber
Titanium Aluminide (35 v/o SiC fibers)	0.0042	-0.0007
Nickel Aluminide (35 v/o W fibers)	0.0013	-0.0005
Nickel Aluminide (30 v/o Saphikon fibers)	0.0014	-0.0004

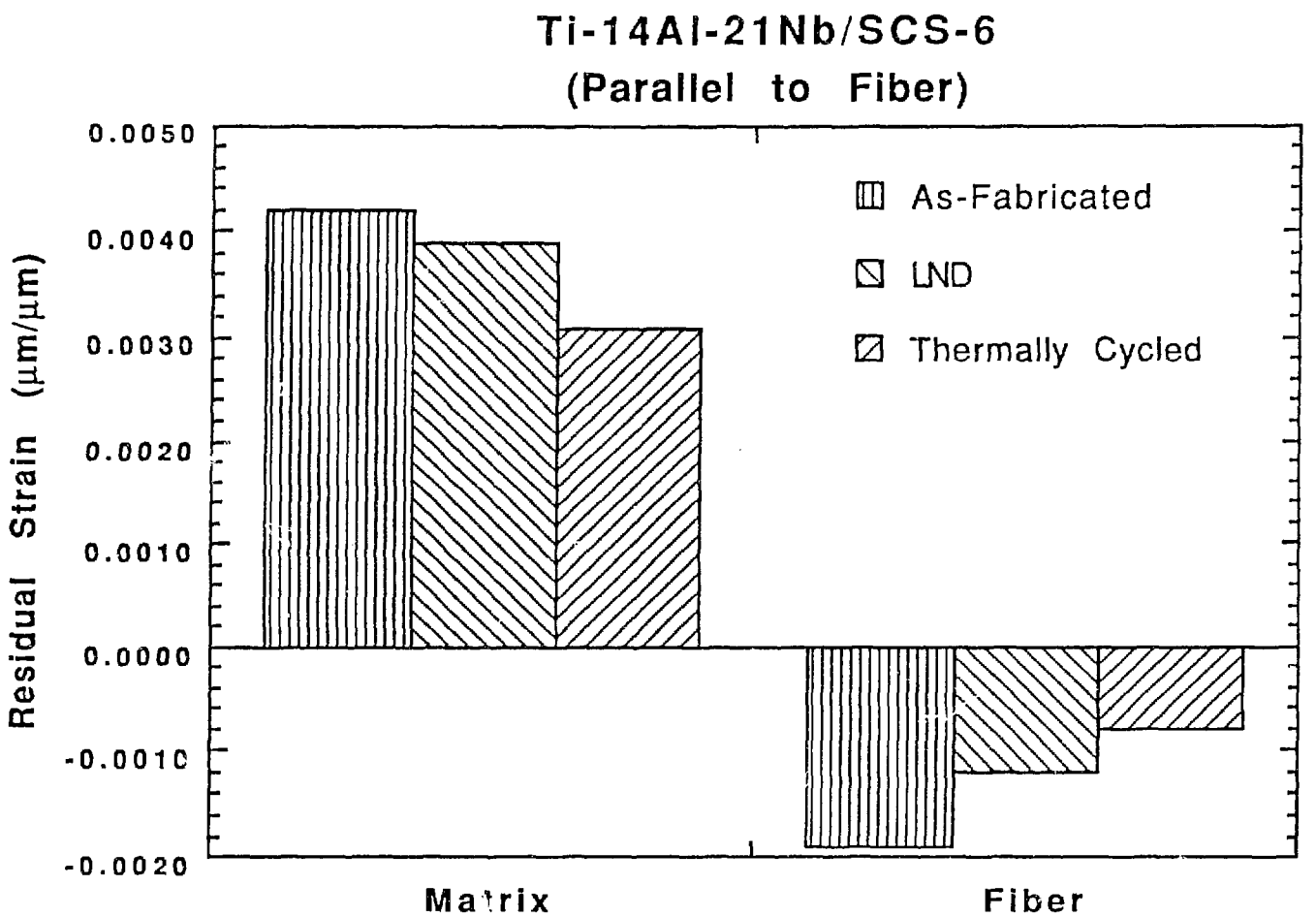
FIGURES


Incident
Neutron
Beam
Lattice Spacing Measured
Perpendicular to Fiber Direction
from 90°-L Detector
Diffracted Neutron Beam 90°-L
L
Matrix
Fiber
Lattice Spacing Measured
Parallel to Fiber Direction
from 90°-R Detector
R
Diffracted Neutron Beam 90°-R


0.005
0.003
0.001
-0.001
-0.003
-0.005
Residual Strain (μm/μm)
0
200
400
600
800
1000
Temperature (°C)
Ti-14Al-21Nb Matrix
(1.75 Å Line Parallel to Fiber)
SCS-6 Fibers
(1.54 Å Line Parallet to Fiber)


Expansion Parallel to
Fiber Axis (Detector 69)
Ti-14Al-21Nb
SCS-6 (220)
Δa / a
1.6 10-2
1.2 10-2
8.0 10-3
4.0 10-3
0.0 100
0
200
400
600
800
1000
1200
Temperature (°C)


Expansion Perpendicular to
Fiber Axis (Detector 49)
Ti-14Al-21Nb
SCS-6 (220)
Δa / a
1.6 10-2
1.2 10-2
8.0 10-3
4.0 10-3
0.0 100
0
200
400
600
800
1000
1200
Temperature (°C)


Ti-14Al-21Nb/SCS-6
(Parallel to Fiber)
Residual Strain (μm/μm)
0.0050
0.0040
0.0030
0.0020
0.0010
0.0000
-0.0010
-0.0020
As-Fabricated
LND
Thermally Cycled
Matrix
Fiber