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## Abstract

Neutron diffraction was used to measure residual thermal strains developed during postfabrication cooling in titanium aluminide and nickel aluminide intermetallic matrix composites. Silicon carbide/Ti-14Al-21Nb, tungsten and sapphire/NiAl, and sapphire and SiC-coated sapphire /$NiAl_{25}Fe_{10}$ composites were investigated. The thermal expansion coefficient of the matrix is usually greater than that of the fibers. As such, during cooldown, compressive residual strains are generated in the fibers and tensile residual strains are generated in the matrix, parallel to the fibers. Liquid-nitrogen dipping and thermal cycling tend to reduce the fabrication-induced residual strains in silicon carbide-fiber-reinforced titanium aluminide matrix composites. However, matrix cracking can occur as a result of these processes. The axial residual strains in the matrix were lower in the nickel aluminide matrix than in the titanium aluminide matrix. As the matrix undergoes plastic deformation, residual thermal strains are related to the yield stress of the matrix.

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# Introduction

Silicon carbide–fiber–reinforced titanium matrix composites, along with tungsten and sapphire–fiber–reinforced nickel aluminide matrix composites, are currently being evaluated for structural applications in jet engines and propulsion systems because of their low weight, high strength, and high stiffness potential at high temperatures. These composites are usually fabricated at temperatures around 1000°C. As there is 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). In addition, subsequent mechanical behavior of high–temperature composites is strongly influenced by the process–induced residual strains and stresses that are locked in the constituents during cooling after fabrication. Therefore, it is important to be able to measure and estimate the residual strains and stresses that exist in composite materials.

A number of programs have been conducted to measure residual strains and stresses in composite materials. For crystalline structures, including crystalline constituents in composite systems, one of the most widely used method involves X–ray diffraction. X–rays have been used to determine microstresses in systems with relatively light elements, such as aluminum/silicon carbide composites (3). However, X–rays typically measure stress fields in the near-surface region. Neutron diffraction is another powerful tool for measuring bulk elastic residual strains, from which residual stresses can be calculated in much the same way as done with X–ray diffraction (4,5). Neutron diffraction offers significant advantages over X–ray diffraction. Neutrons can penetrate deep into the interior of most engineering materials, typically by a factor of 1000 deeper than can X–rays. This provides for an excellent volume sample, a true bulk measurement of 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). Krawitz et al. measured residual stress as a function of temperature in a high volume fraction tungsten carbide–nickel cemented carbide composite (7). Saigal and Kupperman have used neutron diffraction to measure residual thermal strains in NiAl matrix composite systems (8). In addition, Majumdar et al. have used the Intense Pulsed Neutron Source (IPNS) and the General Purpose Powder Diffractometer (GPPD) at Argonne National Laboratory to measure residual strains in a number of engineering composite materials (9).

In the present study, the neutron diffraction technique was used to measure and compare residual thermal strains, developed during cooling, in silicon carbide–fiber–reinforced titanium aluminide, tungsten and sapphire–fiber–reinforced nickel aluminide (NiAl), and sapphire and SiC coated sapphire-fiber-reinforced nickel aluminide ($NiAl_{25}Fe_{10}$) high–temperature composites. The effect of thermal processing, such as liquid–nitrogen dipping and thermal–cycling, on residual strains in $SiC/Ti_3Al$ composites was 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 be applied to neutrons as follows:

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

where $d_{hkl}$ is the lattice spacing, $2\theta$ is the angle between the incident and the scattered neutron beams when a Bragg peak is detected, $\lambda$ is the de Broglie wavelength of the neutron, and $hkl$ are the Miller indices of the diffracting planes.

In neutron diffraction, the lattice spacings in various crystallographic directions of stress–free powders and/or fibers (which are used to fabricate the composite) are determined first. 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.

The data were collected using the IPNS and the GPPD at Argonne. During a single measurement at IPNS, many diffraction peaks, i.e., crystallographic directions, of each phase are recorded simultaneously in various spatial directions(9). Residual strains in the fibers and the matrix, parallel and perpendicular to the fibers, are measured simultaneously by aligning the fibers at an angle of $45^0$ to the neutron beam and analyzing neutron diffraction data with detectors $\pm 90^0$ from the neutron beam direction, as shown in Fig. 1.

Three different composite systems were investigated. They consist of:

1. 35 volume percent silicon carbide (SCS-6) fibers in a Ti-14Al-21Nb ($Ti_3Al$) matrix. Columnar grains of the 140 μm diameter fibers were oriented normal to the fiber axis.

2. 35 volume percent tungsten and 30 volume percent sapphire fibers in nickel aluminide (NiAl) matrix. The tungsten fibers and sapphire fibers were 140 μm and 200 μm in diameter, respectively.

3. 17 volume percent sapphire and 10 volume percent SiC coated sapphire fibers in nickel aluminide ($NiAl_{25}Fe_{10}$) matrix. The fibers were 150 μm in diameter.

## Results and Discussions

Lattice parameters for various crystallographic directions were measured parallel and perpendicular to the fiber axis in the different composite systems. For the silicon carbide-fiber-reinforced titanium aluminide ($Ti_3Al$) samples, the diffraction peak that provided the most useful data for silicon carbide fibers was that for the {220} plane. For the titanium aluminide matrix, the 1.75 Å line provides the average residual strain in the matrix. In order to reduce residual strains and stresses in the composites as compared to those in the as-fabricated condition, 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 2 compares the measured residual strains in the as-fabricated, liquid-nitrogen-dipped, and thermally cycled (100 times to 650°C) silicon carbide-fiber-reinforced titanium aluminide samples. In the as-fabricated condition, at room temperature and parallel to the fibers, the residual strain in the fibers is compressive (-0.0019) and that in the matrix is tensile (+0.0042). The tensile strains in the matrix and compressive strains in the fibers are lower in the LND and thermally cycled specimens than in the as-fabricated sample. Thermal cycling appears to reduce the residual strains more than LND. The transverse strains in the fiber and matrix are both compressive (-0.0005 and.- 0.0007, respectively). It was found that the matrix and fiber strains perpendicular to the fibers did not change significantly with processing.

Based on figure 2, there is no doubt that the axial strains (parallel to fibers) in the fibers and the matrix are reduced by the processing methods discussed. The limited ductility of the matrix is not sufficient to produce the observed reductions in residual strains. It is possible that matrix cracking occurs because of thermal processing; this is further suggested by elastic moduli measured by the velocity of ultrasound propagating perpendicularly 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 1 shows the measured residual strains in the matrix in various composites. The residual strains (parallel to the fibers) are tensile in the matrix and are higher in the titanium aluminide matrix than in the nickel aluminide matrix. The strains in the nickel aluminide (NiAl) matrix due to 35 vol.% tungsten fibers and 30 vol.% sapphire fibers are similar. Based on 3-D elastoplastic finite element analyses, the thermally induced effective stress in the matrix exceeds the yield stress and the matrix undergoes plastic deformation (8,10). This suggests that the residual matrix strains are primarily controlled by the yield stress of the matrix. For the composite systems investigated, it was found that as the yield stress of the matrix increases, the axial residual strain in the matrix increases.

## Conclusions

Neutron diffraction was used to measure residual thermal strains in titanium aluminide and nickel aluminide intermetallic matrix high-temperature composites. It has been shown that significant residual strains develop in these composites during postfabrication cooling. As the thermal expansion coefficient of the matrix is usually greater than that of the fibers, the fibers are under compressive residual strains and stresses. Liquid-nitrogen-dipping and thermal cycling tend to reduce the fabrication-induced residual strains in the silicon carbide-fiber-reinforced titanium aluminide matrix composite. However, matrix cracking can occur as a result of these processes. Strains in the matrix (parallel to fiber axis) are tensile and are higher in the titanium aluminide matrix than in the nickel aluminide matrix. As the matrix undergoes plastic deformation, the residual matrix strains are primarily controlled by the yield stress of the matrix. For the composite systems investigated, it was found that as the yield stress of the matrix increases, the axial residual strain in the matrix increases.

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Table 1 Residual strains in the matrix (parallel to the fibers)

| | Residual strain in the matrix (±0.0003) | Yield stress of the matrix (MPa) |
|---|---|---|
| Titanium Aluminide<br>(35 vol.% SiC fibers) | +0.0042 | 620 |
| Nickel Aluminide<br>($NiAl_{25}Fe_{10}$)<br>(17 vol.% sapphire fibers) | +0.0022 | 414 |
| Nickel Aluminide<br>($NiAl_{25}Fe_{10}$)<br>(10 vol.% SiC-coated sapphire fibers) | +0.0017 | 414 |
| Nickel Aluminide (NiAl)<br>(30 vol % sapphire fibers) | +0.0014 | 240 |
| Nickel Aluminide (NiAl)<br>(35 vol%. tungsten fibers) | +0.0013 | 240 |

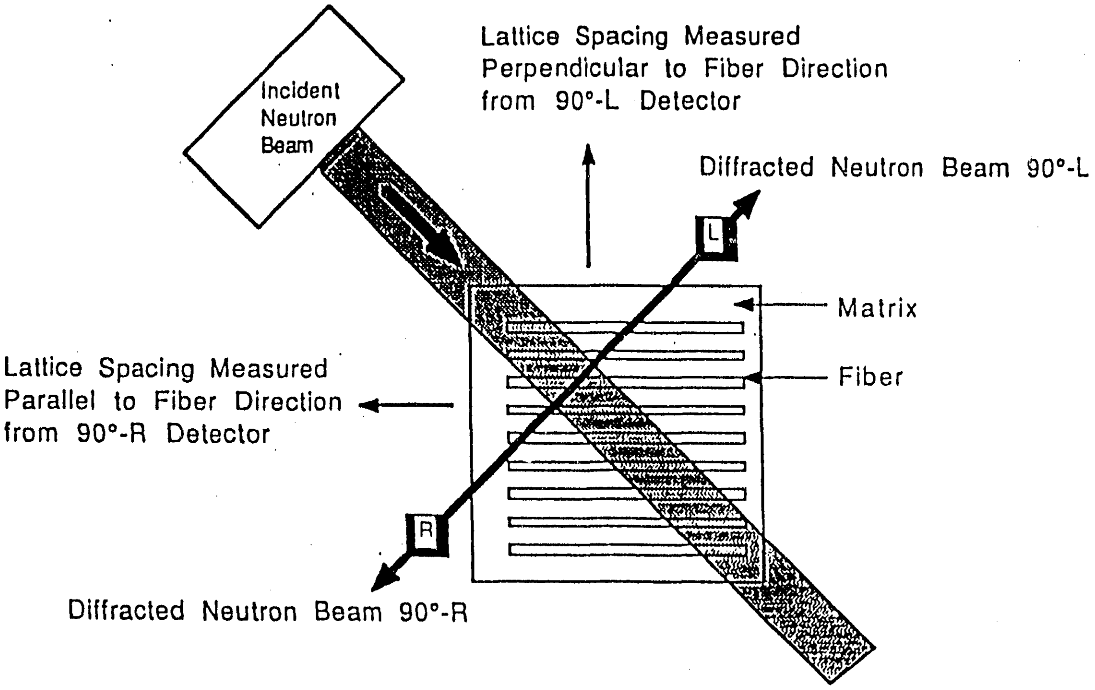

Figure 1. Schematic representation of experimental set-up.

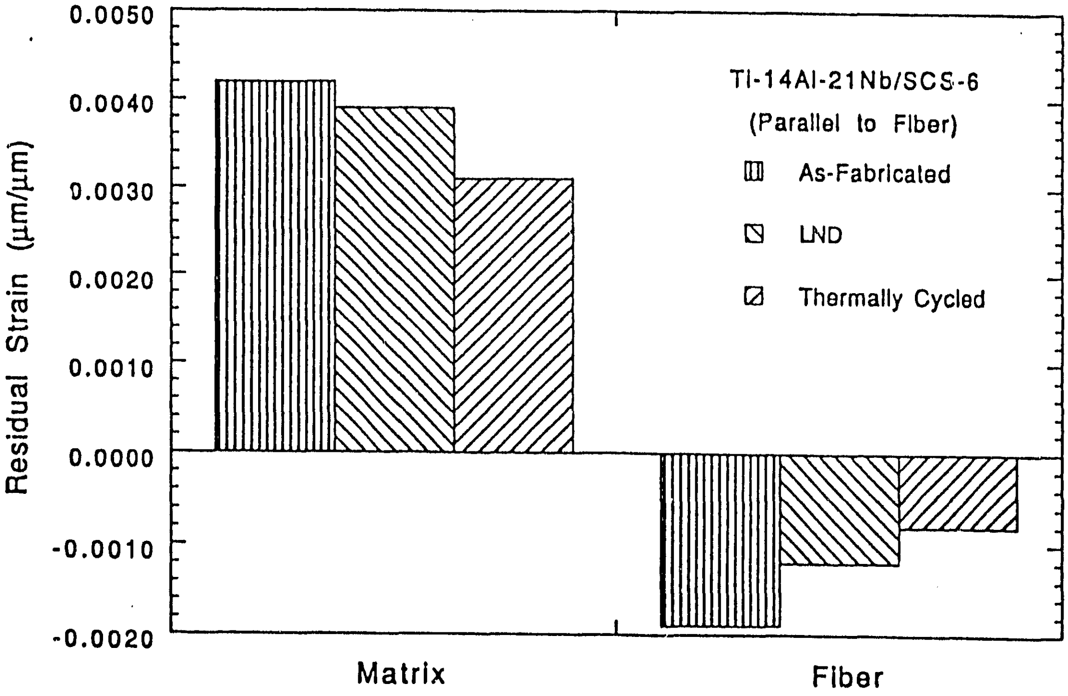

Figure 2. Residual strains in silicon carbide (SCS-6) fibers and titanium aluminide (Ti-14Al-21Nb) matrix, parallel to fiber axis, in as-fabricated, liquid-nitrogen-dipped, and thermally cycled specimens.

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