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TITLE: TEXTURE AND RESIDUAL STRAIN IN SIC/TI-6-2-4-2
TITANIUM MATRIX COMPOSITES

AUTHOR(S): P. Rangaswamy, MST-8
M. Daymond, ISIS, Rutherford-Appleton lab
M.A.M. Bourke, MST-8
R. VonDreele, LANSCE 12
K. Bennett, LANSCE 12
J.A. Roberts, LANSCE 12
N. Jayaraman, Univ. of Cincinnati

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Texture and residual strain in SiC / Ti-6-2-4-2 Titanium Matrix Composites

¹ Partha Rangaswamy*, ² Mark Daymond, ¹ M. A. M. Bourke, ¹ R. Von Dreele, ¹ K. Bennett,

¹ J.A. Roberts, ³ N. Jayaraman

¹ Lujan Center, H805

Los Alamos National Laboratory

Los Alamos, New Mexico 87545

USA

² ISIS, Rutherford Appleton Laboratory

Chilton, Didcot

Oxon, OX11 0QX

England

³ Department of Materials Science and Engineering

University of Cincinnati

Cincinnati, Ohio 45221-0012

USA

* Email: partha@lanl.gov

<http://www.lansce.lanl.gov/mlnsc>

Abstract:

Residual strain and texture variations were measured in two Titanium matrix composites reinforced with Silicon Carbide fibers (Ti/SiC) of similar composition but fabricated by different processing routes. Each composite comprised a Ti-6242 α/β matrix alloy containing 35% by volume continuous SiC fibers. In one composite, the matrix was produced by a plasma spray (PS) route, and in the other by a wire drawing (WD) process. The PS and WD composites were reinforced with SCS-6 (SiC) and Trimarc (SiC) fibers, respectively. The texture in the titanium matrices differed significantly, from approximately $\approx 1.1\times$ random for the monolithic and composite produced by PS route to $\approx 17\times$ random in the monolithic and $\approx 6\times$ random in the composite produced by the WD route. No significant differences in matrix residual strains between the composites prepared by the two procedures were noted. The Trimarc (WD) fibers recorded higher ($\approx 1.3\times$) compressive strains than the SCS-6 (PS) fibers in all the measured directions. The plane-specific elastic moduli, measured in load tests on the un-reinforced matrices, showed little difference.

1. Introduction:

Titanium matrix composites (TMCs) reinforced with continuous silicon carbide (SiC) fibers have received considerable attention in the past three decades. They have potential to replace conventional titanium and nickel-base alloys in aerospace systems such as advanced turbine engines and hypersonic vehicles [1,2], where high specific strength and stiffness at elevated temperatures are critical. However, one concern in their development is the presence of residual stress and its influence on fracture stress, toughness, fatigue resistance and other mechanical properties. The mismatch in the thermal expansion coefficients between titanium and SiC combined with fabrication temperatures around 900°C causes residual stresses to develop during processing. For some TMCs, estimates of the matrix residual stresses are as much as 75% of the yield strength [4]. These may cause localized matrix-yielding around the fiber/matrix interface in ductile systems or cracking in brittle ones.

Despite extensive characterization [4-7] of the effect of residual stresses on TMC mechanical behavior, measurement and modeling of residual stresses is a growing challenge. New processes result in more complicated or highly textured microstructures. Owing to the elastic and plastic anisotropy of the hexagonal lattice of the titanium alpha (α) phase, preferred orientation plays an important role in determining the mechanical behavior in titanium alloys containing a significant α volume fraction [3].

Models that predict residual stresses and their effect on lifetime predictions have to be validated. Therefore, it is important for residual stresses to be experimentally determined and texture or mechanical anisotropy must be accounted for in order to achieve reliable design

parameters. Conversely, if the anisotropy is understood, it can possibly be exploited to offer economic and performance benefits.

This study demonstrates characterization of texture and strain in two TMCs using time-of-flight (TOF) neutron diffraction. Results are compared for two processes, which result in the titanium matrix having a random and strong texture [6,18].

2. Application of Neutron Diffraction:

2.1: Problem of X-ray Diffraction for Textured Materials.

X-ray and neutron diffraction techniques are both viable for measuring strain or texture in TMCs because of the polycrystalline nature of the constituents. However, X-ray measurements are restricted to near surface regions [9-15] which in some circumstances may not be representative of the interior [14].

The majority of measurements are reported for systems that have random or mild texture. "Strong" texture in the matrix hinders the application of the conventional X-ray " d vs. $\sin^2\psi$ " approach since some diffraction peaks may have insufficient intensity at some ψ tilts to obtain accurate d -spacings. Indeed, a strong texture may also impede monochromatic neutron measurement, since it may be hard to satisfy a particular monochromatic (wavelength) setting coupled with a high angle diffraction peak (though tilting is not a prerequisite) [19].

2.2: Pulsed Neutron Diffraction

The Lujan Center at the Los Alamos National Laboratory is a pulsed source of spallation neutrons. It is a national user facility available to scientists from industry, academia, and other national laboratories for condensed matter experiments. Due to the polychromatic nature of the

neutron beam at a pulsed source all possible lattice reflections are recorded subject to texture and the scattering geometry.

2.3: Pulsed Neutron Diffraction for Strain Measurements using the Neutron Powder Diffractometer (NPD).

Neutron diffraction is performed routinely on the NPD at Los Alamos National Laboratory [13,14,19]. Due to the good penetration of neutrons, these are bulk measurements. Since strains are measured from many different hkl's, the technique is equally convenient for textured and untextured samples. One advantage of having multiple peaks recorded is a more comprehensive description of the deformation than if a single family of grains were examined, corresponding to one Bragg reflection [13,18]. Moreover, all the lattice reflections are recorded with the same resolution in each spectrum, and multiphase materials are conveniently examined.

2.4: Pulsed Neutron Diffraction for Texture Measurements using the High Intensity Powder Diffractometer (HIPD) [8].

HIPD is used for powder diffraction studies of texture, magnetic materials, small samples, or polycrystalline materials subjected to pressures up to 10 GPa. For texture measurements a collection of complete diffraction patterns for 4 to 6 different scattering directions are taken simultaneously at a set of predetermined orientations (usually 10-20) of the sample to the incident neutron beam. This procedure yields 50-70 diffraction patterns which are distributed over the possible angular orientation space. These diffraction data are subsequently analyzed by a Rietveld refinement procedure which uses a unified spherical harmonics description of the variation in the reflection intensities due to texture [8,21,22]. The refined values of the spherical harmonic coefficients are then used to generate complete pole figures.

3.0 Sample Preparation:

3.1: Plasma Sprayed Composites

Plasma sprayed (PS) TMCs were fabricated at Textron Specialty Materials [8]. In the plasma spray process (figure 1b), superheated droplets of Ti-6242 matrix are deposited on silicon carbide fibers (SCS-6) wound on the surface of a rotating and axially translated drum. Advantages include the virtual elimination of fiber swimming (the fiber is rigidly held in place by sprayed metal), no added ribbon or wire is required and almost any matrix can be selected (the process uses metal powder as a starting material). The reinforcing 142 μm dia. SiC fibers (SCS-6) were also produced by Textron Specialty Materials, Inc. The fiber was produced by the Chemical Vapor Deposition (CVD) deposition of SiC onto a carbon monofilament $\sim 35 \mu\text{m}$ in dia. and the subsequent deposition of $\sim 6\mu\text{m}$ of carbon in a 3-layered outer coating. The plasma sprayed fiber monotape and foil were cut into panels. These were stacked for fabrication of the TMC [0]₁₀ using a hot-isostatic-press (HIP) technique (1010°C/100MPa/2 hours). Metallography of the plasma sprayed matrix of the composite revealed an equiaxed $\alpha + \beta$ microstructure. The matrix had about 90 % alpha (HCP) and 10% beta (BCC) as measured using X-ray diffraction. The fiber volume fraction (v_f) was 35%. The fiber has been shown to have a face centered cubic (fcc) crystal structure (Space group F -4 3 m, lattice parameter $a \approx 4.360 \text{ \AA}$) [24].

3.2: Wire Drawn Composites

Wire drawn (WD) composites were produced by Atlantic Research Corporation using Ti-6Al-2Sn-4Zr-2MO cold drawn wire (diameter 152/178 μm) and Trimarc-1(SiC) fibers (diameter 127 μm , figure 1a). The Ti-6242 cold drawn wire was obtained from a commercial weld-wire source according to AMS specification 4975-F. The SiC fibers were obtained from Americom

(produced by their joint venture with 3M). The fiber was produced by the chemical vapor deposition (CVD) of SiC onto a tungsten monofilament and the subsequent deposition of $\sim 3\mu\text{m}$ of carbon in a 3-layered outer coating and had a fcc crystal structure similar to SCS-6. The circular mat was slit across a drum, yielding a large, rectangular mat. Panels were cut, bagged, outgassed (to remove the binder), then Hot Isostatically Pressed (HIP) at a nominal cycle of $966^\circ\text{C}/100\text{MPa}/2.2\text{hours}$. The panels were subsequently given a matrix age heat treatment of $593^\circ\text{C}/8\text{hours}$. Specimens of $100\text{ mm} \times 25\text{ mm} \times 2.5\text{ mm}$ were taken from these panels by water jet cutting, then diamond ground. The composite samples were 10 ply, with a nominal volume fraction of fibers being 35%. Although similar to the PS matrix, the matrix had a little less beta, comprised of 95% alpha (HCP) and 5% beta (BCC) as measured using X-ray diffraction.

Residual strain measurements for both the PS and WD matrices were restricted to the alpha (α) phase. Since the low volume fraction of beta phase made it difficult to obtain a diffraction pattern for the beta phase.

4. Texture Measurements on High Intensity Powder Diffractometer (HIPD) :

Samples of the monolithic matrix and composites measuring $20\text{ mm} \times 50\text{ mm} \times 2.2\text{ mm}$ thick were used for both texture and strain measurements. For the texture measurements the High Intensity Neutron Diffractometer (HIPD) was used [8]. Each sample was mounted on a two-circle (ω, χ) goniometer with the fiber direction perpendicular to the incident neutron beam. The sample was rotated in each of the 13 selected orientations and diffraction data collected for approximately 45 min. In addition, by using six of the detector banks on HIPD, eight different diffraction vectors were measured at each orientation setting. This yielded 78 diffraction patterns

for subsequent analysis by a Rietveld refinement procedure, using a spherical harmonics description of the texture in General Structurel Analysis System (GSAS) [20,21]. Pole figures were then computed from the refined values of the harmonic coefficients.

Figure 2 (a,b,c) show the pole figures for the prism plane (100), basal plane (001) and the pyramid plane (111) for the PS matrix material in the composite, for the WD monolithic and for the WD composite matrices. The pole figures for the PS matrix show an almost completely random texture of 1.1 x random. By contrast, the WD matrix exhibits a strong fiber texture with the normal to the prism planes (100) oriented in the drawing direction, and consequently a strong texture of the (001) planes transverse to the drawing direction. Note that the texture in the composite matrix was reduced to 6 x random compared to the neat matrix texture of 17 x random. One reason for the reduction could be the annealing of the matrix material during the composite fabrication process where temperatures of 965 °C and isostatic pressures of 100 MPa are applied for two hours. This may induce recovery and re-orientation of some of the grains during flow of the matrix material around the fibers.

5. Residual Strain Measurements on Neutron Powder Diffractometer

In a diffraction experiment the direction in which strain is measured is defined by the orientation of the scattering vector with respect to the sample. With the fibers oriented at 45° to the beam, the four detectors located at $2\theta = \pm 90^\circ$ and $\pm 148^\circ$ provide simultaneous strain measurements at 0°, 29°, 61° and 90° to the fiber axes [18]. Measurements were made in two orientations (figure 3, a-b). In the first orientation (figure 3a) detectors at ± 90 degrees measure the longitudinal $\langle \epsilon_{LL} \rangle_{\text{Long}}$ and through thickness $\langle \epsilon_{NN} \rangle_{\text{Long}}$ strains. In the second orientation (figure 3b) the transverse $\langle \epsilon_{TT} \rangle_{\text{Trans}}$ and $\langle \epsilon_{NN} \rangle_{\text{Trans}}$ through thickness strains were recorded. The

second orientation was adopted to verify that the strains were transversely isotropic (which they were). The irradiated volume was $\sim 840 \text{ mm}^3$ and count periods of ~ 6 hours at a proton beam current of $70 \mu\text{A}$ were used.

Bragg reflections were fitted individually and strains (ϵ_{hkl}) in the composites were calculated by comparison to the measurements in the monolithic alloys or fibers by ;

$$\epsilon_{hkl} = (d_{hkl} - d_o)/d_o \quad (1)$$

where d_{hkl} and d_o are the interplanar spacings in the composite and unstressed standards, respectively. Figure 4(a,b) shows diffraction patterns for both PS and WD samples. The PS sample showed all possible reflections possible while in the WD sample the strong texture was apparent. Only well defined and distinct reflections were used for strain calculations. The reflections 002,102,101,112,201,110,100 were examined in the matrix, representing the basal, prism, pyramid and other planes. In the fibers in both the PS and WD the 220 and 440 reflection was used for strain determination. Plane-Specific-Elastic-Strains (PSES) for both composites were calculated using equation 1 and are shown in Table 1.

5.1 Strain measurements in Plasma Sprayed TMC

Figures 5a and 5b are plots of measured strain for the orientations shown in the longitudinal and transverse directions for the PS TMC. In figure 5a the strains for the PS TMC are plotted against the angle α from 0 to 90° where, $\alpha = 0^\circ$ and $\alpha = 90^\circ$ are parallel and perpendicular to the fiber axis respectively. In seven diffracting planes representing prism (100), basal (002), pyramidal planes (101), and others (110), (102), (112), and (201) were examined. In the longitudinal orientation the highest strains were recorded for $\alpha = 0$ and lowest strain values for $\alpha = 90$ for any set of crystallographic planes in the matrix. Except for the basal plane (002)

and the pyramid plane (102) the strains for all the examined reflections were within 500 $\mu\epsilon$ of one another for both the longitudinal (figure 5a) and transverse (figure 5b) orientation of the fibers. The strain in the basal plane (002) was the only dramatic outlier, with a variation of 400 $\mu\epsilon$ at $\alpha = 0^\circ$, increasing to 1450 $\mu\epsilon$ at $\alpha = 90^\circ$ (figure 5a), from the mean of all the other planes. In the transverse orientation (i.e., fiber axis vertical to the incident neutron beam) the PS material showed little variation in strains with varying β angles ($\beta = 0^\circ$ and $\beta = 90^\circ$ are perpendicular to the fibers in the transverse and normal directions respectively). For the fibers, similar trends as observed for the matrix are seen for the longitudinal and transverse orientations, except that the strains are compressive.

5.2 Strain in the Wire Drawn TMC

In figure 6a the strains for the WD TMC are plotted against the angle α from 0 to 90° . Due to the strong 'fiber' texture in the matrix, only prism planes of the $\langle 100 \rangle$ type were observed at $\alpha = 0^\circ$. For $\alpha = 29, 57$ and 90° , prism (110) and pyramidal planes (101,102,112,201) produced diffraction peaks. Variation in the strains for the (100), (200), and (300) prism planes (which should of course be identical) were within $\pm 3 \%$ of one another at approximately 3500 $\mu\epsilon$. For $\alpha = 90^\circ$ the strains range from + 48 to + 2219 ($\mu\epsilon$), with the prism plane (110) showing the lowest value and the basal planes (002) showing the highest value.

In the transverse orientation (figure 6b) all diffracting planes were noted at each angle of β . This was expected since the WD matrix fiber texture was parallel to the fiber direction. Like the PS TMC, in the transverse orientation the strains for diffracting planes do not show significant variation with changing angle of β (figure 6b). Reassuringly, the values in figure 6b

are similar at $\beta = 90^\circ$ are similar to the strains measured in the longitudinal orientation (figure 6a) at $\alpha = 90^\circ$.

In the longitudinal orientation (figure 6a) the trend of decreasing residual strains with increasing α angle is similar to that observed in the PS matrix. The fibers also show trends similar to the PS matrix for the longitudinal and transverse orientations, except that the strains are slightly higher in compression.

6. Plane-Specific-Elastic-Constants (PSEC) measurements on NPD.

Plane-specific elastic constants for the monolithic WD and PS matrices were determined using the in-situ loading capability on the neutron powder diffractometer [34]. These results are tabulated for both WD and PS monolithic matrix materials in Table 2. The results are also compared with calculations of plane specific elastic constants made using stiffness constants S_{11} , S_{12} , S_{13} , S_{33} , and S_{44} for pure single crystal titanium [25]. Surprisingly, there was close agreement between the measured polycrystalline values and the calculated single crystal values.

7. Discussion:

7.1 Merits of Neutron Diffraction

The objective of this research was to demonstrate the use of a pulsed neutron source to characterize a wire drawn TMC for texture and strain and to compare the results with a plasma sprayed TMC. Pulsed neutron diffraction strain measurement in composite materials has many advantages over other techniques such as monochromated neutrons, X-ray, ultrasonic, matrix dissolution etc.) [27-33]. This advantages include : 1) The entire diffraction spectrum with all possible reflections is available, offering a selection of reflections for calculating strains. 2) The availability of the entire diffraction spectra makes the technique invulnerable to strain measurements in even strongly textured materials, as at least a single or several reflections can be

found in any particular scattering geometry. 3) Since strains for several reflections are available the ambiguities sometimes associated with strains measured from single reflection is lessened. 4) The use of Rietveld profile refinement techniques can provide average lattice parameters from which an empirical bulk average strain can be estimated [26]. 5) Since neutrons penetrate deep into the material (this also applies to monochromated neutrons) mean phase strains in composites (fiber and matrix) can be measured.

In previous studies of titanium alloy composites the matrix material has typically exhibited random or mild texture [13-18]. However, the actual texture has not been documented. Many of the previous studies were on composites fabricated by the foil fiber foil process, where the matrix material is in the form of foils measuring few hundred microns in thickness [1-2, 4]. When the conventional d vs. $\sin^2\psi$ X-ray technique was used to determine stresses, we can infer that the texture was not strong enough to hinder diffraction peaks from appearing at all angles of the ψ tilt of the sample. The measurement of residual strain in the strongly textured wire drawn matrix composite was achieved efficiently and provides a direct comparison with the results for texture free composite.

7.2 Strain Comparison between WD and PS matrices.

In figures 5 and 6 the plane specific strains for the PS and WD matrices show a spread at each value of α or β but the values are similar for comparable hkl's. The plane specific strains for the PS and WD matrices are plotted together in figure 7 for the longitudinal orientation of the fibers. Only the hkl's (002) basal, (100), (110) prism, (101) and (201) pyramid planes are selected for providing clarity in reading the figure 7, and these hkl's are representative of the range in the strains as shown in figures 5 and 6. From the figures the plane specific strains for the different hkl's show a spread that is lowest parallel to the fiber direction (0° to the fiber) and highest

perpendicular to the fiber direction. We also observe, that the (100) and (110) prism planes show the least strain and the (002) basal planes show the highest strains. A similar trend exists for the WD matrix, except that there are few planes that appear at more than two angular locations so comparisons are less precise.

From a previous study [20] we have shown that the hkl strains shown in figure 7 fit the equation $\langle \epsilon_{\alpha} \rangle_{f,m} = \langle \epsilon_{11} \rangle_{f,m} \cos^2 \alpha + \langle \epsilon_{22} \rangle_{f,m} \sin^2 \alpha$, where f and m are fiber and matrix respectively, and $\alpha = 0^\circ$ and $\alpha = 90^\circ$ are parallel and perpendicular to the fiber. We have used this equation to fit all the hkl strains for the PS matrix and also fit the (101) strains of the WD matrix, despite the fact that strains are available at only three angular locations (as against four). The fits (dashed lines) to the (101) strains for the PS and WD matrix show identical trends and similar strains at all the four angular locations.

To capture the comparison between PS and WD in a more quantitative fashion the plane specific strains of the WD and PS matrices are re-plotted in figure 8a with the plane specific strains of the PS matrix as an independent variable and that of the WD matrix as a dependent variable. If the strain magnitudes are the same for the respective hkl's between the WD and PS matrix material, the slope of the line fit to the WD strains should have a slope of 1 and essentially the two lines would be merged together, in practice it is 1.077. The differences in strains are within 10 % at all angular orientations except for two outliers at the 90° orientation, the (112) and (110) hkl's where the % difference was 67 and 91 %, respectively. However, the strains are also small in that particular direction. This suggests that there is more scatter in the direction perpendicular to the fiber.

Within experimental errors and allowing for the possible difference in the SiC reinforcement, these results surprisingly show that there is no significant difference in the matrix

hkl strain distributions between the WD and PS TMCs despite the dramatic difference in texture states !

Figure 8b shows a similar plot of the equivalent hkl plane specific fiber strains for the PS and WD TMCs. Unlike the similar plane specific strains measured in the WD and PS matrices, the plot shows disparity in the strains for the respective hkl's at all the four angular locations. However, the slope of the line fit to the WD strains is 1.13, which is slightly higher than the line fit to the matrix WD strains of 1.077. This suggests that despite the PS TMC's showing lower compressive strains in comparison with the strains observed in the textured TMCs, qualitatively the trends are similar at all the four angular locations. The differences in the strains is not entirely surprising due to the SiC fiber in the PS matrix having a higher elastic modulus (420 GPa) compared to the SiC fiber modulus in the WD matrix (360 GPa) [3-4,6,22]. Also, we cannot neglect the effects of differences in the fiber manufacturing process and differences in the constituents present in the fibers.

In both the PS and WD TMCs the similarity of strains for the different hkl in the transverse orientations indicates transverse isotropy and validates such assumptions used in continuum models for strain or stress predictions. In order to fully understand these observations a complete analysis of the three dimensional stress and strain state of both TMCs has to be conducted.

8. Summary and Conclusions:

Texture and residual strain variations were measured in two TMCs of the same composition but fabricated by a wire drawn and plasma spray processes.

- The texture in the wire drawn monolithic and composite Ti matrices differed significantly.

Texture measurements in the wire drawn monolithic and composite matrices revealed $\approx 17 \times$

random and $\approx 6 \times$ random fiber texture, respectively, with the fiber axis pointing in [100] direction of the HCP unit cell and transversely isotropic. The plasma spray monolithic and composite matrices exhibited random texture.

- Comparison of the tensile plane specific matrix residual strains found in the WD and PS TMCs showed no significant differences. In both cases transverse isotropy was observed.
- Residual strains in the fiber were similar and compressive for both TMCs. However, the fibers in the WD TMCs showed a higher ($\approx 1.3 \times$) compressive residual strains than the PS TMCs.

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Table 1

Comparison of Plane Specific Elastic Strains ($\langle \epsilon \rangle_{hkl}$) for Wire drawn and Plasma Sprayed Matrices

HKLs	$\langle \epsilon \rangle_{hkl}, \mu\epsilon$ (Wire-drawn)	$\langle \epsilon \rangle_{hkl}, \mu\epsilon$ (Plasma-Sprayed)
($\equiv$) Parallel to fiber		
100, 200, 300	3451, 3530, 3451	3524
110		3550
201		3681
112		3548
101		3615
102		3747
002		4029

29 ° to fiber		
100		
110	2761	2828
201		3087
112		2861
101	3152	2953
102		2999
002		3441

(61 °) to fiber		
100		
110		1254
201	1118	1471
112		1125
101	1379	1468
102		1845
002		2627

($\perp$) Perpendicular to fiber		
100		
110	48	558
201		843
112	643	383
101	1025	813
102		1402
002	2219, 2258	2109

Table 2

Comparison of Plane Specific Elastic Constants from experimental measurements and from analytical predictions

HKLs	E_{hkl} - Texture (Wire-drawn) (GPa)	E_{hkl} - Untexture (Plasma - Sprayed) (GPa)	E_{hkl} - Single Crystal predictions* (GPa)
100	110	111	103
002	148	140	145
101	116	111	108
110		112	104
112		114	109
201	110	108	104

*Note: This are based on the five independent stiffness constants S_{11} , S_{12} , S_{13} , S_{33} , S_{44}

(a)

(b)

Figure 1. Schematic of the (a) Wire Winding technique and (b) RF Induction Plasma Deposition (Plasma Spray) technique used in fabricating silicon carbide fiber reinforced titanium metal matrix composites.

Bulk Texture Pole figures of Titanium Matrix in Composite Plasma Sprayed Vs. Wire Drawn

Plasma Spray

Wire Drawn

Figure 2: Pole figures for 100, 001 and 111 poles for the matrices of the Plasma Sprayed and Wire Drawn Titanium Matrix Composites.

Figure 3. Schematic of the two orientations in which the samples were placed in the neutron beam; a) longitudinal Strain, $\langle \epsilon_{LL} \rangle_{Long}$ and $\langle \epsilon_{NN} \rangle_{Long}$ b) Transverse Strain measurements $\langle \epsilon_{TT} \rangle_{Trans}$ and $\langle \epsilon_{NN} \rangle_{Trans}$

(a)

(b)

Figure 4. Diffraction plots for the monolithic Ti6242 matrix - a) Plasma Sprayed b) Wire Drawn (scattering vector in the drawing direction).

Figure 5(a,b). Measured hkl strains in the Plasma Sprayed TMC orientations as shown.

(a)

(b)

Figure 6(a,b). Measured hkl strains in the Wire Drawn TMC orientations as shown.

Figure 7. Comparison of selected hkl strains measured in the matrix of WD and PS composites for the fiber orientation shown.

(a)

(b)

Figure 8. Comparison of the hkl specific strains in the WD and PS composite a) matrices and b) fibers.