

# ULTRASONIC EVALUATION OF THE PROCESSING OF FIBER-REINFORCED METAL-MATRIX COMPOSITES

S. Krishnamurthy

*Materials and Processes Division, UES Inc., 4401 Dayton-Xenia Road, Dayton, Ohio 45432-1894, USA*

T. E. Matikas, P. Karpur

*Research Institute, University of Dayton, 300 College Park, Dayton, Ohio 45469-0127, USA*

&

D. B. Miracle

*Air Force Wright Laboratory, Materials Directorate, Wright-Patterson AFB, Ohio 45433-7817, USA*

(Received 14 April 1994; revised version received 29 November 1994; accepted 8 March 1995)

## Abstract

*Non-destructive evaluation techniques using ultrasonic shear wave and longitudinal wave interrogations have been employed to evaluate the consolidation behavior of a Ti-14Al-21Nb/SiC model composite containing a single SiC fiber. Partially and fully consolidated composite samples prepared by diffusion bonding methods were examined by these techniques, and the ultrasonic images obtained correlated with the results of metallographic characterization. A similar evaluation was carried out on a Ti-6Al-4V/SiC single-ply composite to illustrate the applicability of these techniques to composites containing high fiber volume fractions. The results indicate that ultrasonic NDE provides a quick and reliable tool for monitoring the consolidation of metal-matrix composites.*

*Keywords:* non-destructive evaluation, metal-matrix composite, consolidation, ultrasound

## 1 INTRODUCTION

Several methods are currently available for the fabrication of metal-matrix and intermetallic-matrix composites. In particular, composites based on reactive matrices with high melting temperatures such as titanium alloys are usually processed by solid state diffusion bonding of matrix foils, powders, or sprayed deposits with reinforcements. For example, the processing of continuously reinforced titanium-matrix composites by the foil-fiber-foil method typically involves diffusion bonding of rolled matrix alloy foils with reinforcing fibers in the form of woven mats with a cross-weave to hold the fibers in place. The foils and

the fiber mats are stacked alternately and consolidated by vacuum hot pressing or hot isostatic pressing. The processing conditions are carefully selected in order to achieve complete consolidation and produce acceptable composite material. While higher temperatures and longer processing times may enable consolidation, they can promote undesirable reactions at the fiber/matrix interface and also cause high residual stresses after the composite is cooled to ambient temperature. On the other hand, lower processing temperatures can lead to fiber damage and incomplete consolidation. In practice, optimum processing conditions are determined from preliminary diffusion-bonding experiments on small samples. The initial processing temperatures are chosen on the basis of known flow characteristics of the matrix alloy at different temperatures and strain rates, and the consolidation of these samples is checked by metallographic examination of polished sections. However, the use of metallography alone is generally inadequate since consolidation often occurs non-uniformly within the composite panels.

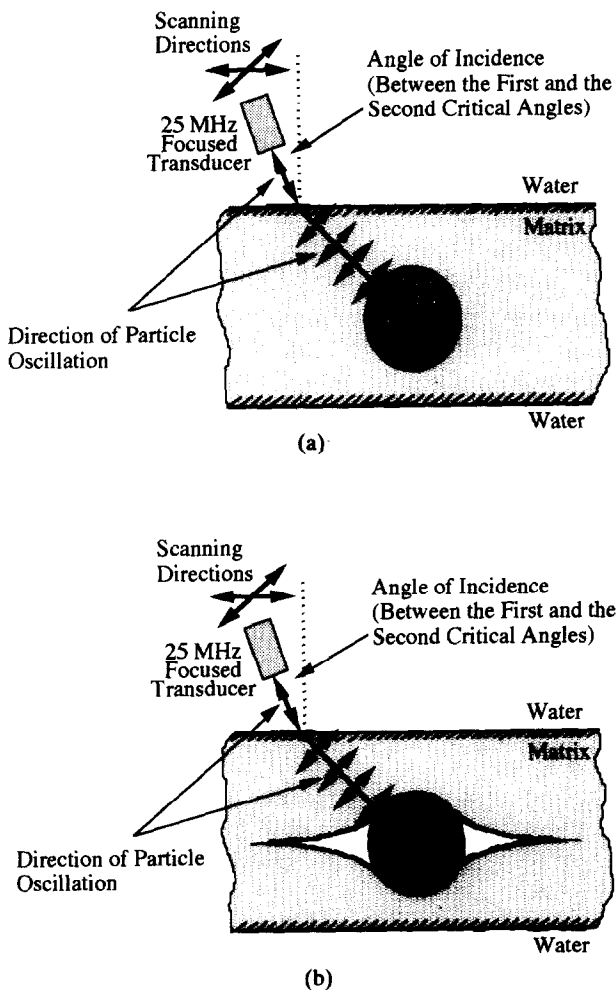
The use of non-destructive evaluation (NDE) techniques for characterizing the quality and mechanical behavior of composite materials is well known. Specifically, ultrasonic NDE techniques have been used in the past for determining the distribution of reinforcements and detecting the defects present in composite panels.<sup>1</sup> In the case of fiber-reinforced metal-matrix composites (MMCs), ultrasonic methods have been employed to screen for macroscopic defects such as ply delaminations and non-uniform fiber spacing arising from either missing fibers or

displacement of fibers during fabrication.<sup>1,2</sup> The objective of this communication is to demonstrate that ultrasonic NDE is an equally valuable tool for detecting microscopic defects arising during the fabrication of composites and that it can be reliably used to minimize the number of iterations required for optimization of the consolidation process. The application of two different ultrasonic techniques involving shear wave and longitudinal wave interrogations for evaluating the consolidation of two model titanium-alloy composites will be discussed.

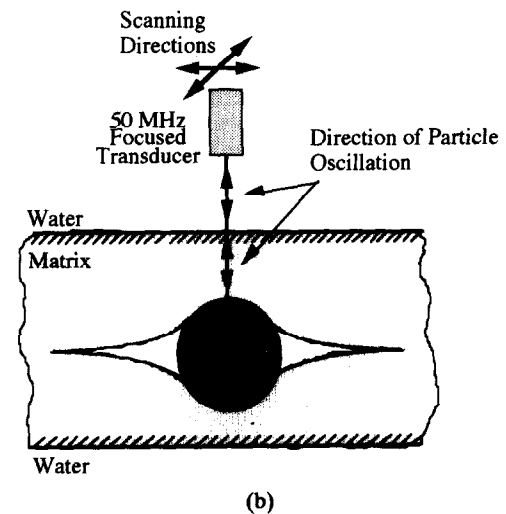
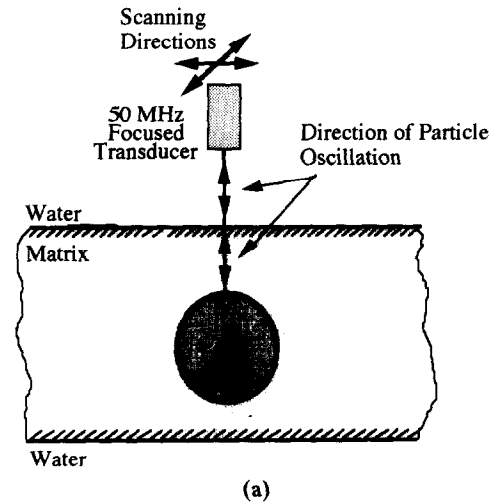
## 2 EXPERIMENTAL APPROACH

### 2.1 Fabrication of single-fiber composites

These model composite samples consisting of an SCS-6 SiC fiber in a Ti-14Al-21Nb (wt%) alloy matrix were fabricated in the form of 2 mm thick panels by diffusion bonding together two matrix alloy sheets with a single fiber between them. Two different



**Fig. 1.** Schematic diagram of the transverse section of a continuously reinforced composite showing shear wave interrogation of a fiber embedded in the matrix: (a) fully consolidated composite, and (b) partially consolidated composite.



**Fig. 2.** Schematic diagram of the transverse section of a continuously reinforced composite showing longitudinal wave interrogation of a fiber embedded in the matrix: (a) fully consolidated composite, and (b) partially consolidated composite.

processing conditions were used; (1) vacuum hot pressing at 925°C under 5.5 MPa pressure for 30 min followed by hot isostatic pressing (HIPing) at 1010°C under 100 MPa pressure for 2 h (Panel A), and (2) vacuum hot pressing at 982°C under a pressure of 9.2 MPa for 30 min (Panel B).

### 2.2 Fabrication of single-ply composites

The single-ply composites consisted of a layer of SCS-6 SiC fibers in a Ti-6Al-4V (wt%) alloy matrix. These were also fabricated in the form of 2 mm thick panels by vacuum hot pressing at 954°C under a pressure of 9.2 MPa for 2 h (Panel B).

### 2.3 Ultrasonic imaging

First, the procedure used for evaluating the Ti-14Al-21Nb/SiC single-fiber composite samples will be described. The fiber embedded in the matrix

material was ultrasonically imaged by two different techniques: (1) shear-wave interrogation and (2) longitudinal-wave interrogation. Figures 1 and 2 show schematic illustrations of these two techniques. In the shear-wave technique, a 25 MHz focused transducer (6.3 mm diameter, 12.7 mm focal length) was used in the pulse-echo mode. The ultrasonic wave front was incident on the specimen surface inclined to the vertical plane at an angle of either 18 or 24°. Both of these angles lie between the first and the second critical angles, which are defined as the angles of incidence above which longitudinal and shear waves, respectively, will not propagate in the matrix material. As a result, only vertically polarized shear waves propagated in the matrix and were incident on the fiber/matrix interface (Fig. 1). In the longitudinal wave technique, a 50 MHz focused transducer (6.3 mm diameter, 25.4 mm focal length) was used in the pulse-echo mode and the wave front was incident normal to the specimen surface (Fig. 2). Under this condition, a compressional wave propagates in the matrix and is reflected from the fiber back to the transducer. The reason for using a transducer of a different frequency for the longitudinal wave technique is as follows. The wavelength of the wave propagating in the matrix is calculated by the simple expression,  $\lambda = c_i/f$ , where  $c_i$  is the velocity of the ultrasonic stress wave ( $c_i$  is denoted by  $c_s$  or  $c_L$  depending on the type of wave propagation i.e. a shear or a longitudinal wave, respectively) and  $f$  is the frequency of interrogation. In the case of the Ti-14Al-21Nb matrix alloy, the velocity of the shear wave generated by the 25 MHz transducer was measured to be  $c_s = 3209$  m/s and, therefore, the wavelength was about 130  $\mu$ m. The velocity of the longitudinal wave propagating in the same matrix was measured to be  $c_L = 6489$  m/s, which is approximately twice the shear wave velocity. Since the frequency of longitudinal wave interrogation was twice that of shear wave interrogation, the wavelength remained the same (about 130 microns). Consequently, the two interrogation techniques can be directly compared for the same resolution in terms of wavelength. In both of these techniques, the image of the fiber was obtained by scanning the ultrasonic transducer along and across the fiber with an increment of 25  $\mu$ m between signal acquisition points. At each point, the back-reflected ultrasound was software-gated for imaging as discussed elsewhere.<sup>3</sup>

The ultrasonic techniques were also used for evaluating the Ti-6Al-4V/SiC single-ply composite specimens. The measured ultrasonic shear and longitudinal wave velocities in the Ti-6Al-4V matrix alloy were found to be  $c_s = 3163$  m/s and  $c_L = 6141$  m/s, respectively. It should be noted that the elastic properties of the matrix material (Ti-6Al-4V) used for the single-ply composites are close to the

properties of the matrix material (Ti-14Al-21Nb) used for the single-fiber composites. Therefore, the ultrasonic velocities in the matrix are practically the same and the wavelength of ultrasound remains the same (about 130  $\mu$ m). However, because of the small spacing between fibers in the single-ply composite, a smaller angle of incidence (19°) was used in order to image the fibers without overlapping.

The angles of incidence of the transverse modes internal to the sample corresponding to different angles of incidence were calculated using Snell's law of mode conversion and are listed in Table 1.

In order to study the correlation between the ultrasonic image and the local microstructure, all the composite specimens were sectioned normal to the fiber axis at several locations along the fiber. These sections were metallographically polished and examined by optical microscopy.

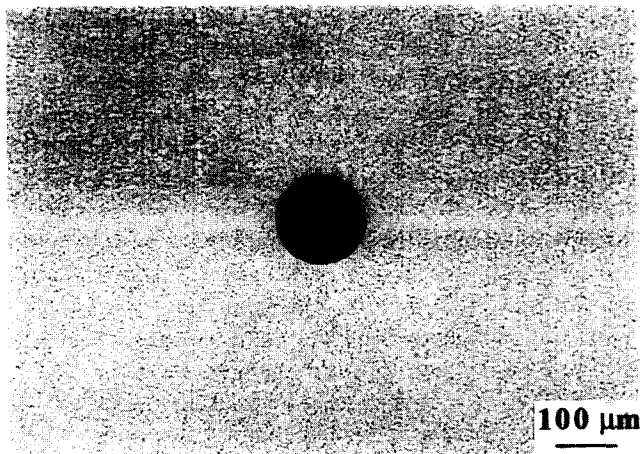
### 3 RESULTS AND DISCUSSION

The ultrasonic images of the Ti-14Al-21Nb/SiC single-fiber composite sample which was processed by vacuum hot pressing followed by HIPing (Panel A) are shown in Fig. 3. Figure 3(a) corresponds to shear wave interrogation and shows uniform reflection along the fiber. Figure 3(b) shows the image resulting from longitudinal wave interrogation of this material and indicates that the reflected signal is uniformly weak along the length of the fiber. Fig. 4 shows two ultrasonic images obtained by shear wave interrogation of the composite sample which was consolidated by vacuum hot pressing alone (Panel B). Both these images, which were recorded with different angles of wave front incidence, show significant variations in the reflected intensity along the length of the fiber. In fact, very little reflection is observed from significant lengths of the fiber. These features indicate non-uniformity in this panel which is also evident from the image obtained by longitudinal wave interrogation (Fig. 5).

An optical micrograph from a cross section of the composite panel A is shown in Fig. 6. This micrograph shows that the consolidation of the matrix around the fiber is complete in this composite. This result was

**Table 1. Ultrasonic shear wave mode conversion angles ( $\theta_i$ ) in the two titanium-alloys at different angles of incidence ( $\theta_s$ )**

Matrix alloy	Mode conversion angle ( $\theta_s$ )		
	$\theta_i = 18^\circ$	$\theta_i = 19^\circ$	$\theta_i = 24^\circ$
Ti-14Al-21Nb	42°	44.9°	61.9°
Ti-6Al-4V	41.3°	44.1°	60.4°

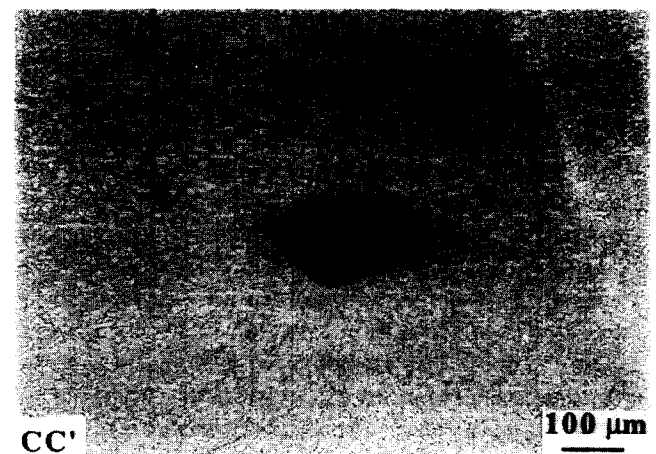
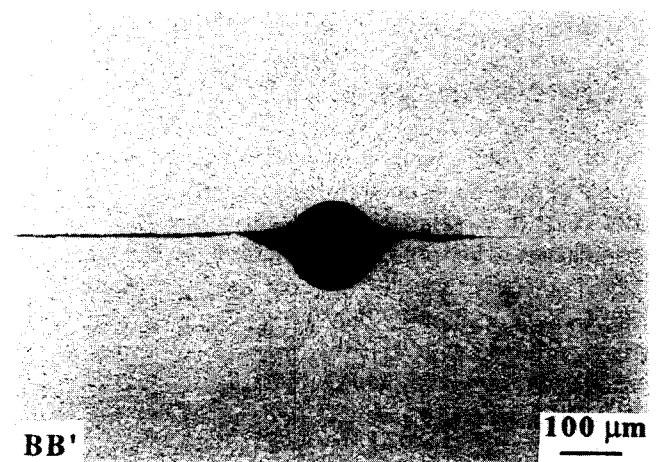
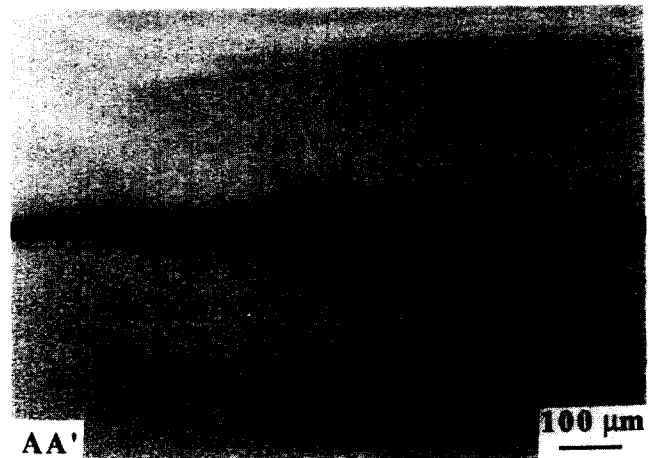


**Fig. 6.** Optical micrograph of Ti-14Al-21Nb/SiC single-fiber composite (Panel A) sample showing good consolidation.

typical of the various cross-sections examined, suggesting that the consolidation occurred uniformly within panel A. Figure 7 shows optical micrographs taken from the composite panel B corresponding to the sections AA', BB' and CC' which are indicated in Fig. 5. It is clear from these micrographs that the bonding of the matrix alloy sheets is incomplete around the fiber. Further, there is a considerable variation in the degree of bonding of the matrix along the length of the fiber (Fig. 7).

A good correlation is found between the microstructure of the Ti-14Al-21Nb/SiC single-fiber composite panels and the observed ultrasonic images. These results show that the diffusion-bonding conditions used for processing of panel B did not lead to complete consolidation, whereas panel A was fully consolidated. These results are also in general agreement with previous observations dealing with the sequence of events during the consolidation of such composites by the foil-fiber-foil method.<sup>4,5</sup> When the foil-fiber-foil preform is subjected to elevated temperature under pressure, the process begins with the indentation of matrix alloy foils by the fibers and is followed by matrix creep, leading to diffusion bonding between the foils. At the end of this stage, two large pores remain on opposite sides of each fiber along the bond plane of the foils due to lower local stress<sup>4,5</sup> as observed in Fig. 7. The composite is fully consolidated when these remnant voids near the fibers are eliminated (Fig. 6). The present results also indicate that consolidation can occur non-uniformly along the length of the fiber (Fig. 7). Clearly, metallographic characterization alone is tedious under such conditions and other techniques such as ultrasonic NDE will be very useful.

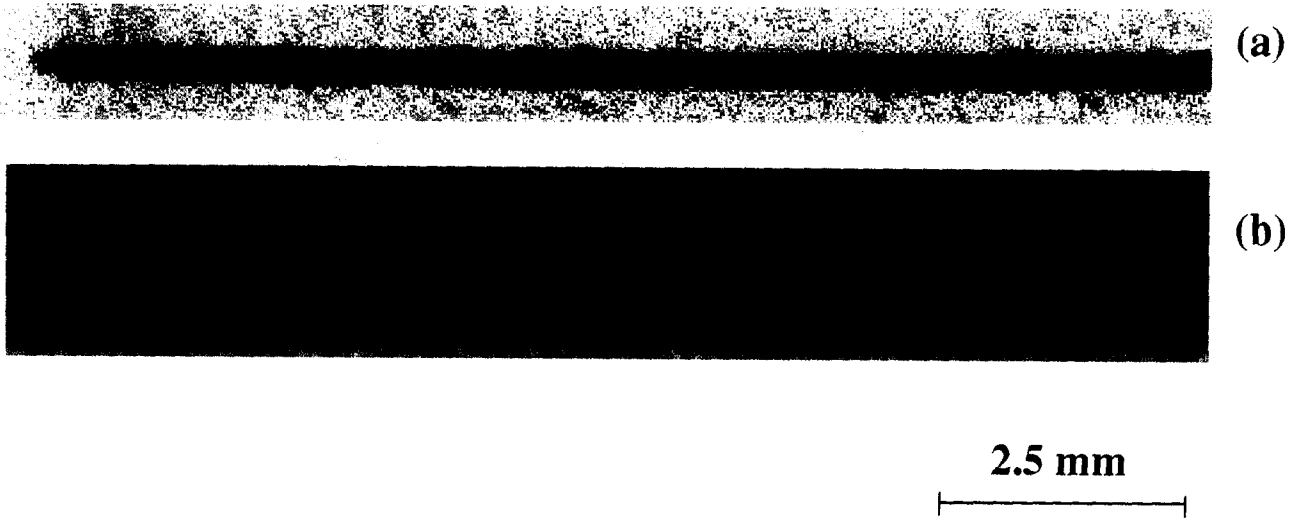
The ultrasonic images are formed when ultrasonic energy is reflected from the scattering cross-section of a reflector (cylindrical fiber) present in the path of



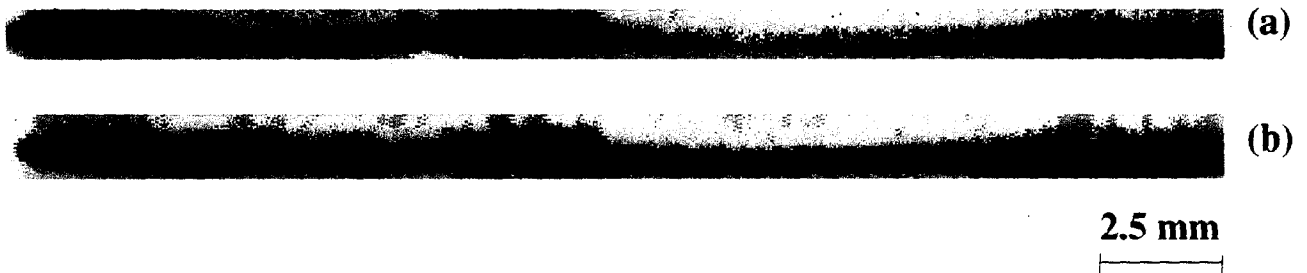
**Fig. 7.** Optical micrographs of vacuum hot pressed Ti-14Al-21Nb/SiC (Panel B) single-fiber composite sample showing poor consolidation at sections AA', BB', and CC', respectively, which are indicated in Fig. 5.

wave propagation through a homogeneous medium (matrix). In the present application, the matrix may be assumed to be homogeneous since the wavelength of interrogation ( $130\ \mu\text{m}$ ) is large compared to the average grain size of the matrix ( $<10\ \mu\text{m}$ ).

In the case of shear wave interrogation, mode-converted shear waves propagate through the matrix and are back-scattered from the fiber (Fig. 1). When



**Fig. 3.** Ultrasonic image from Ti-14Al-21Nb/SiC single-fiber composite which was consolidated by vacuum hot pressing + HIPing (Panel A): (a) shear wave interrogation with a wave front incidence of  $24^\circ$ , and (b) longitudinal wave interrogation with normal incidence.



**Fig. 4.** Ultrasonic images from Ti-14Al-21Nb/SiC single-fiber composite (Panel B) using shear wave interrogation with different angles of wave front incidence: (a)  $18^\circ$  and (b)  $24^\circ$ .

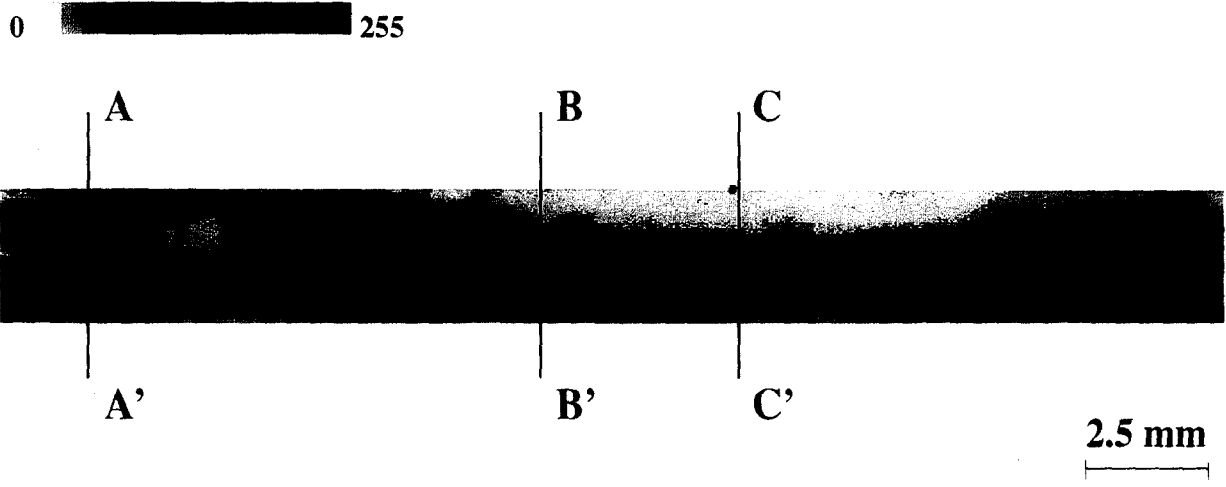


Fig. 5. Ultrasonic image from Ti-14Al-21Nb/SiC single-fiber composite (Panel B) using longitudinal wave interrogation; AA', BB', and CC' indicate sections at which metallographic samples were examined.

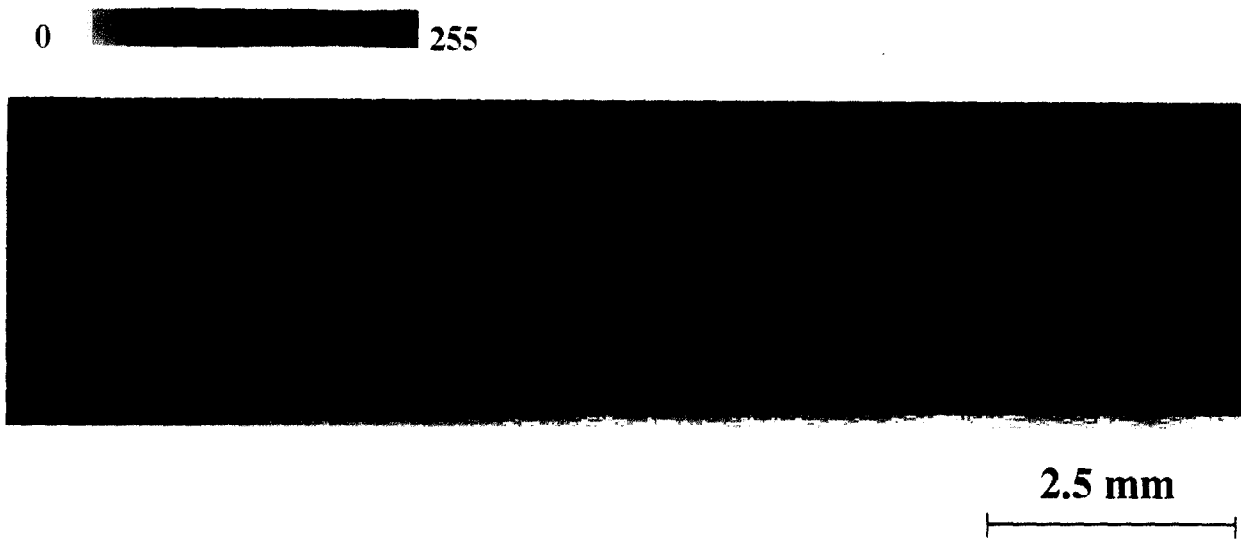


Fig. 8. Ultrasonic image from a Ti-6Al-4V/SiC single-ply composite using shear wave interrogation showing uniform reflection on the left side and non-uniform distribution of reflected intensity in the middle and right side regions.

the matrix is completely consolidated around the fiber, the maximum of the reflected signal occurs when the polarized shear waves propagating in the matrix are incident perpendicular to the ruled surface of the cylindrical fiber as indicated in Fig. 1(a). It should be noted that the maximum of the received energy corresponds to the main lobe of the Bessel function which is the theoretical response of a cylindrical reflector embedded in a medium.<sup>6</sup> Thus, a well-consolidated sample will provide an image as shown in Fig. 3(a). On the other hand, when the composite is poorly consolidated, the reflector is defined by the envelope that includes both the fiber and the void regions. In other words, the reflector boundaries are the matrix/fiber boundaries together with the matrix/void boundaries and, therefore, the reflector has a non-cylindrical shape (Fig. 1(b)). In this case, the reflected signal can have either low or high amplitudes owing to shear wave scattering from matrix void regions which usually have variable curvatures. For a given angle of incidence of ultrasonic waves on the surface of the composite panel, the angle of refraction of the shear waves propagating in the matrix is defined according to Snell's law.<sup>7</sup> By changing the angle of incidence, the effect of the slope of the matrix/void boundary can be evaluated and the optimum angle of incidence corresponding to the maximum ultrasonic signal from that boundary may be determined. The use of different angles of incidence as shown in Fig. 4 is thus necessary in order to determine the extent of the void. Furthermore, because of the variable slope and hence different 'effective scattering cross-section' of the reflector, the image of the fiber appears to possess a variable diameter along its length. This implies that the void shape varies along the length of the fiber as a result of non-uniform consolidation.

In the case of longitudinal wave interrogation, compressional waves are incident normal to the surface of the panel specimen as well as the foil-foil interface as shown in Fig. 2. When the composite is

fully consolidated, longitudinal waves are much less sensitive to various fiber/matrix interfacial conditions as compared to shear waves,<sup>8</sup> and this should result in poor image delineation. As Fig. 3(b) shows, the small dynamic range under these conditions makes it very difficult to distinguish between the fiber signal and the background noise from the matrix. It should be noted that although the image shown in Fig. 3(b) was obtained after signal processing, the reflected amplitude does not show significant fluctuations. However, the normal incidence longitudinal waves are sensitive to any lack of consolidation or delamination between the matrix alloy foils or sheets and will produce high reflected ultrasonic amplitudes which can be mapped to outline these zones as observed in Fig. 5.

Although these results were obtained from a model composite containing a single fiber, these ultrasonic techniques are equally applicable for studying the consolidation of real composites containing a high volume fraction of fibers. Figure 8 shows an ultrasonic image obtained by shear wave interrogation of the Ti-6Al-4V/SiC single-ply composite specimen. The non-uniform distribution of reflected intensity along a number of fibers in this panel is evident from this image. As discussed earlier in the case of the single-fiber composite, the region on the left side of this image showing uniform reflection from each fiber appears to be fully consolidated. Metallographic examination of sections from this uniform region confirmed good consolidation as shown in Fig. 9. Metallographic sections corresponding to the middle and right side regions of Fig. 8 with non-uniform reflection revealed incomplete consolidation similar to the results shown in Fig. 7. With regard to the evaluation of composites containing a high volume fraction of fibers, it should be noted that the shear wave technique is useful for studying the outermost layer of fibers while the longitudinal wave interrogation is more suited for detecting macroscopic defects such as voids and delamination.

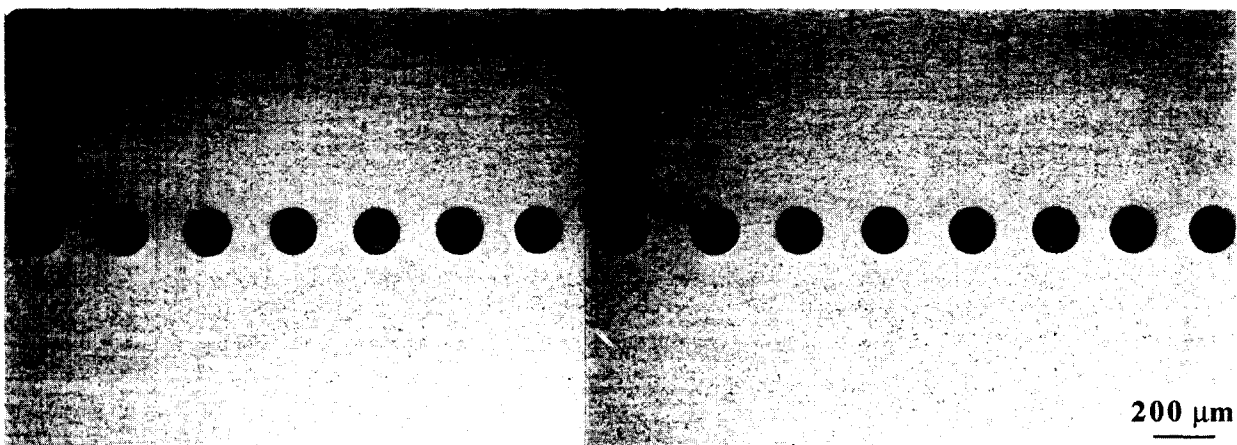


Fig. 9. Optical micrograph showing a transverse section of Ti-6Al-4V/SiC single-ply composite from a well-consolidated region corresponding to uniform reflection in Fig. 8.

In addition to the studies of composite consolidation, these NDE methods are very useful for the detection of fiber fractures and fiber/matrix interfacial debonding in continuously reinforced metal-matrix composites and this has been reported elsewhere.<sup>9</sup>

#### 4 SUMMARY

Two different ultrasonic NDE techniques using shear wave and longitudinal wave interrogations were applied to study the consolidation characteristics of Ti-14Al-21Nb/SiC single-fiber composites and Ti-6Al-4V/SiC single-ply composites. A good correlation was found between the ultrasonic images and the extent of consolidation of the matrix alloy around the fibers as determined by metallography. The results demonstrate the usefulness of these ultrasonic techniques in evaluating the processing behavior of metal-matrix composites.

#### ACKNOWLEDGEMENT

This work was supported by and performed on site in the Materials Directorate, Air Force Wright Laboratory, Wright-Patterson AFB, under Contract Nos F33615-91-C-5663 (S. Krishnamurthy) and F33615-89-C5612 (T. E. Matikas, P. Karpur).

#### REFERENCES

1. Johnson, W. S., Screening of metal matrix composites using ultrasonic C-scans. *J. Comp. Technol. Res.*, **11** (1989) 31-4.
2. Liaw, P. K., Shannon, R. E., Clark, W. G., Harrigan, W. C., Jeong, H. & Hsu, D. K., Determining material properties of metal-matrix composites by NDE. *JOM*, **44** (1992) 36-40.
3. Buynak, C. F., Moran, T. J. & Martin, R. W., Delamination and crack imaging in graphite-epoxy composites. *Mater. Evaluation*, **47** (1989) 438-47.
4. Guo, Z. X. & Derby, B., SiC Fiber/Ti-based composites: processing, microstructure and interface properties. In *Titanium '92: Science and Technology*, ed. F. H. Froes & I. L. Caplan. The Minerals, Metals and Materials Society, Warrendale, PA, 1993, pp. 2633-9.
5. Nicolaou, P. D., Pichler, H. R. & Kuhni, M. A., Fabrication of Ti-6Al-4V matrix, SCS-6 fiber composites by hot pressing using the foil-fiber-foil technique. In *Developments in Ceramic and Metal-Matrix Composites*, ed. K. Upadhyaya. The Minerals, Metals and Materials Society, Warrendale, PA, 1991, pp. 37-47.
6. Schuetz, L. S. & Neubauer, W. G., Acoustic reflection from cylinders-nonabsorbing and absorbing. *J. Acoust. Soc. Am.*, **62** (1977) 513-7.
7. Krautkrämer, J. & Krautkrämer, H., *Ultrasonic Testing of Materials*. Springer-Verlag, New York, 1990.
8. Matikas, T. E. & Karpur, P., Ultrasonic reflectivity technique for the characterization of fiber-matrix interface in metal matrix composites. *J. Appl. Phys.*, **74** (1993) 228-36.
9. Karpur, P., Matikas, T. E. & Krishnamurthy, S., Matrix-fiber interface characterization in metal matrix composites using ultrasonic imaging of fiber fragmentation. *Proceedings of the American Society for Composites 7th Technical Conference*, Pennsylvania State University, University Park, PA, 1992, pp. 420-7.