

# A Study on the Ultrasonic Nondestructive Evaluation of Carbon/ Carbon Composite Disks

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It is desirable to perform nondestructive evaluation (NDE) to assess material properties and part homogeneity because the manufacturing of carbon/carbon brake disks requires complicated and costly processes. In this work several ultrasonic techniques were applied to carbon/carbon brake disks (322mm *od*, 135mm *id*) for the evaluation of spatial variations in material properties that are attributable to the manufacturing process. In a large carbon/carbon disk manufactured by chemical vapor infiltration (CVI) method, the spatial variation of ultrasonic velocity was measured and found to be consistent with the densification behavior in CVI process. Low frequency (e.g., 1-5MHz) through-transmission scans based on both amplitude and time-of-flight of the ultrasonic pulse were used for mapping out the material property inhomogeneity. Images based on both the amplitude and the time-of-flight of the transmitted ultrasonic pulse showed significant variation in the radial direction. The radial variations in ultrasonic velocity and attenuation were attributed to a density variation caused by the more efficient densification of pitch impregnation near the *id* and *od* and by the less efficient densification away from the exposed edge of the disk. Ultrasonic velocities in the edges of the disk. Ultrasonic velocities in the thickness direction were also measured as a function of location using dry-coupling transducers; the results were consistent with the densification behavior. However, velocities in the in-plane directions (circumferential and radial) seemed to be affected more by the relative contents of fabric and chopped fiber, and less by the void content.

**Key Words:** Carbon/Carbon Brake Disk, Pitch Impregnation, Chemical Vapor Infiltration (CVI), Dry-Coupling Transducers, Ultrasonic Testing

## 1. Introduction

Owing to the advantage of very large strength-to-weight and stiffness-to-weight ratios, composite materials (Im, Kim and Yang, 1999) are attrac-

tive for a wide range of applications. Increasingly, more and more high performance engineering structures are being built with critical structural components made from composite materials. Especially, carbon/carbon (C/C) composites are one of the few materials that are suitable for structural applications at high temperature environments (e.g., above 2000°C) (Buckley, 1988) while maintaining strength and stiffness. Also, these composites possess a high resistance to thermal shock and are stronger at an elevated temperature. Thus, in the aerospace and vehicles industry, C/C composite materials are being used for

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aircraft brake disks, space shuttle rocket nozzles, exhaust cones, air inlet channels, connecting rod, engine piston and turbine rotor (Lewis, 1988). One such application is an aircraft brake disk. As compared to steel brakes, carbon/carbon brakes are lighter by about 40% and last twice as long as the steel brakes are in terms of the number of landings per overhaul (Awasthi and Wood, 1988). Aircraft brake manufacturers are therefore making brake disks and rotors out of carbon/carbon composites (Buckley, 1988 and Fitzer, 1987). Aircraft brakes are critical components that serve multiple functions: they are the friction member, the heat sink elements, and the structural elements.

The manufacturing process for C/C composites is rather complex compared with that for conventional composites such as Graphite/Epoxy due to the multiple steps involved in the carbonization and densification cycles, which results in improved density and thermal conductivity. In one manufacturing approach, C/C composites are made by first producing pre-forms of carbon-fiber, which are then densified by a chemical vapor infiltration (CVI) process (Fitzer, 1987 and Provided, 1998). The CVI process is carried out on the C/C composites at high temperature (about 1000°C). The densification is repeatedly carried out until the matrix is so impervious to the vapor that no more carbon can be deposited on the fibers. At this point, the composites are quenched so that the matrix cracks due to thermal coefficient mismatch and thermal gradient methods. Also, under pressure in service, overcrusting and pore blockage may occur due to pressure gradient methods. The material is then heated again and densification is repeated. This process is repeated several times until a desired density is reached. Up to about three densification/carbonization may be required before the desired density is achieved. Porosity and cracks that often occur due to the mass loss and shrinkage of resin during the processing of carbonization have detrimental effects on the material properties of the C/C composites (Ko and Hone, 1992). To ensure product quality and structural integrity, nondestructive evaluation (NDE)

methods (Stimson and Fisher, 1980 and Nam and Seferis, 1992) are needed for inspecting carbon/carbon brake disks and rotors. Ultrasonic testing is capable of revealing material inhomogeneity and internal defects. More importantly, the velocity of ultrasonic waves is related to the elastic stiffness of the material in a direct relationship.

In the evaluation of material properties, ultrasonic C-scan imaging is an effective NDE technique used for material analysis and quality control in the industries. C-scan imaging in its most conventional implementation is used to map variations in ultrasonic echo peak amplitude that occur when scanning across a material part. Ultrasonic C-scan image provides quantitatively a two-dimensional view of a specimen in which differences in image contrast result from the objects interaction with an impinging ultrasonic wave (Hale, Hsu and Adams, 1996). Also, wave velocity is probably one of the most widely used quantities (Hsu and Hughes, 1992). When simply predicting the material property, it is very desirable to measure velocities on the sample surface. Such a method can be used as a nondestructive evaluation technique on structural components. In this work, several ultrasonic NDE techniques were applied in the evaluation of a developmental C/C brake disk. Through-transmission C-scans in an immersion setup based on both the amplitude and time-of-flight of the transmitted ultrasonic pulse were used for qualitative assessment of the material homogeneity in the plane of the disk. Ultrasonic velocity of longitudinal waves propagating in the thickness direction was measured at selected locations using elastomer-faced dry-coupling transducers. To correlate ultrasonic velocity with density variation and microstructures, a series of specimens were cut out along a radial direction for density measurement and microscopy. Ultrasonic pulse echo C-scans were used for detecting material anomalies near the surface of the disk. Finally, ultrasonic velocity and C-scan in the in-plane directions were measured to obtain the influence of fabric, chopped fiber and void for the six cut-out pieces of a C/C disk.

## 2. Experimental Method

### 2.1 Fabrication of C/C composites

The fabrication method for carbon materials is similar to methods used for ceramic processes. Chopped fibers and Acelan fabric of solid particles (primary carbon part) are combined with coal tar pitch, which acts as precursor during the baking, i.e., carbonization treatment. The main disadvantages in the ceramic-like process method of carbon materials fabrication are the mass loss and shrinkage of precursor during a carbonization process. However, using the chopped fibers and coal tar pitch contribute to the reduction of the shrinkage to some degree.

The first step in fabrication deals with the impregnation of carbon fabrics with the coal tar pitch. This is known as the as-cured step of the composites and then producing of molding packers. The composites are then subjected to carbonization, i. e., heat treatment at high temperatures. At this stage, the carbon of the composite increases, referred to as the carbonized composites. In this process, the pitch powder in the composites is pyrolyzed to form a carbonized matrix. The composite, at this stage of first carbonization, is highly porous and inferior in terms of its mechanical properties and density. Graphitization is performed at high temperatures (about 2000°C) to obtain the solid state transformation of metastable non-graphitic carbon into a graphite structure. The carbonization and graphitiza-

tion steps in carbon-carbon composites are repeated two or three times. In order to improve the mechanical properties, it is then subjected to a densification process, in which the pores formed during pyrolysis are densified by using a chemical vapor deposition process.

### 2.2 NDE experiments

The C/C brake disk used in this study provided in 1998 was a ring-shaped disk 17mm thick, 135mm inner diameter, and 322 mm outer diameter. The disk consisted of a stack of cloth plies and chopped fiber layers and was densified by pitch impregnation and also by chemical vapor infiltration (CVI) at the end of fabrication process. All of the immersion scan and most of the dry-coupled velocity measurements were made with the disk intact. Afterwards, six small blocks were cut out along a radius. The dimensions of each block were 12.7×12.7×17mm as shown in Fig. 1. The densities of the blocks were determined from their individual weight and volume and the internal microstructures were examined using an optical microscope.

The through-transmission ultrasonic C-scans were conducted in an immersion tank using a SONIX scanning system shown in Fig. 2. A pair of 5MHz, 6.35mm diameter, unfocused transducers were aligned perpendicular to the disk and driven by a Panametrics 5052 pulser/receiver. The amplitude and time-of-flight of the (first arrival) transmitted pulse were used in generating the amplitude and time-of-flight images. The

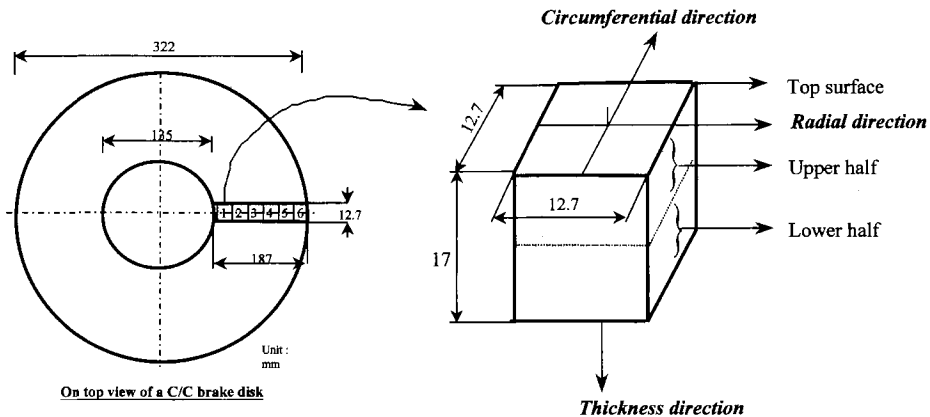


Fig. 1 Dimensions of the brake disk and location of the six small cut-out pieces

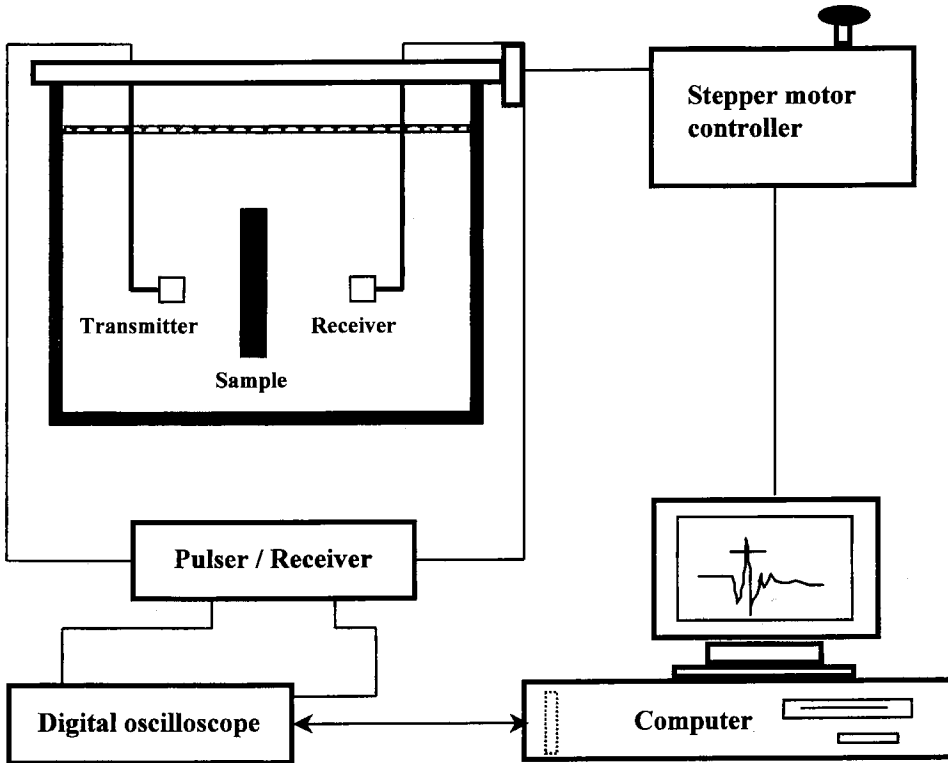


Fig. 2 Experimental setup for through-transmission scan mode in immersion tank

through-transmission measurement sampled the integrated material property in the thickness direction. In the transmission C-scan images, a larger amplitude is usually associated with better consolidated material with a lower attenuation, scattering, or absorption. The recorded time-of-flight in a transmission C-scan represents the sum of that in water and in the sample. Since the water path remains constant, a larger time-of-flight is associated with a lower velocity of sound in the sample. To probe the material properties near the surface, a high frequency transducer (10MHz and 25 MHz, 6.35 mm diameter, unfocused, respectively) was used in the pulse-echo mode. Amplitude C-scans were made using the front surface echo.

### 2.3 Velocity measurement

The ultrasonic velocity in the C/C composite brake disks was measured in the thickness direction, radial direction and circumferential direction specified at several points on the sample using dry-coupling ultrasonics (KD50-1, Ultran

Lab.). All measurements were made with a narrow bandwidth ultrasonic pulse transducers which have high power penetration and high sensitivity because C/C composite brake disk composing the chopped fiber, fabric and pitch makes the ultrasonic transmission difficult, and data were obtained in the time domain. In this work, contact and planar transducers of dry-coupling was used (see Fig. 3).

To obtain a quantitative, accurate value of the ultrasonic velocity, a pulse-overlap method with dry-coupling transducers was used (Hsu, Liawand and Yu, 1994). These transducers contain an elastomer face layer and can be coupled by applying pressure. The measurement setup is shown in Fig. 2. To obtain the ultrasonic transit time through the sample, the difference in transmit time between two ultrasonic pulses was measured. The first pulse was transmitted through the reference piece but without the sample in plane and the record pulse was transmitted through the reference piece plus the sample. The

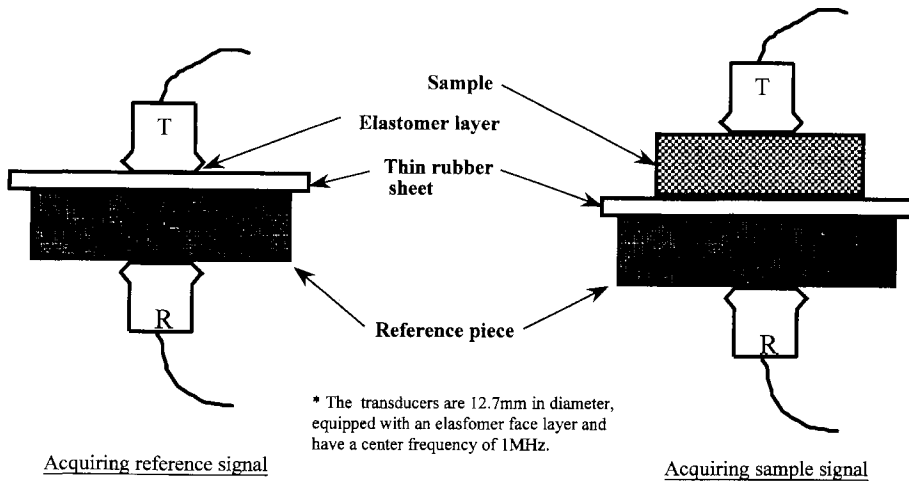


Fig. 3 Velocity measurement method using dry-coupling transducers

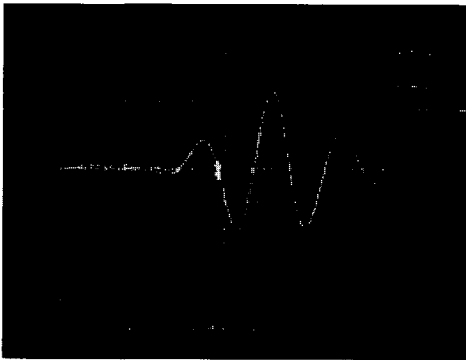


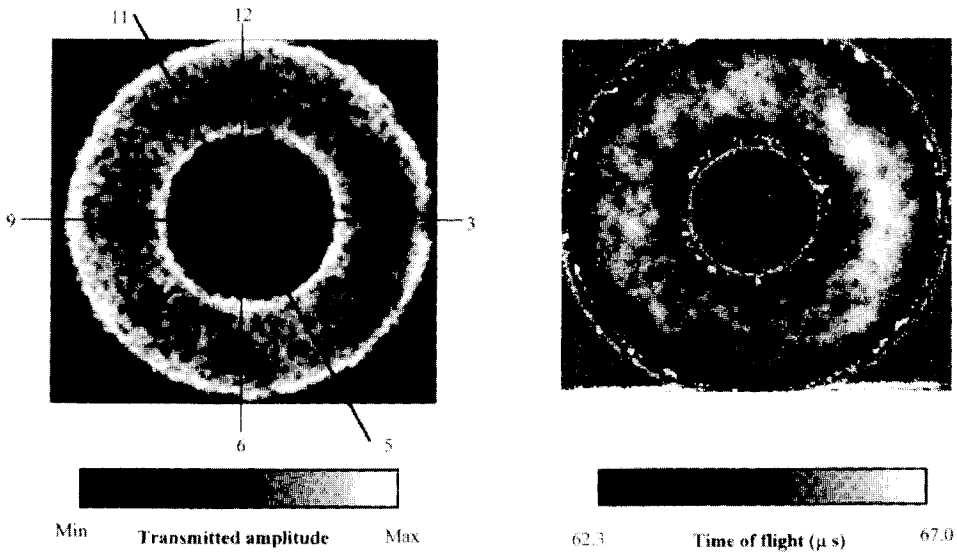
Fig. 4 The pulse-overlap method applied C/C composites

thin rubber sheet was present in both cases to provide dry-coupling between the reference piece and the sample and to ensure that its own transit time was canceled out. These two pulses were stored in the memory of a LeCroy 9400 digital oscilloscope and their difference was obtained by shifting one pulse to overlap and match with the other pulse. The velocity was simply the sample thickness divided by the transit time through the sample. In this experiment, the transducers used were 1MHz, 12.7mm diameter (KD50-1, Ultralan Lab.). The ability to store, shift, expand, invert and magnify the signals on the scope screen proved very convenient for performing the measurements manually. Figure 4 shows the typically overlapping pulses.

### 3. Results and Discussion

#### 3.1 Through transmission images of C-Scans

The amplitude and time-of-flight C-scan images of the through-transmission scan are shown in Fig. 5. The amplitude image showed that the transmitted signal was generally greater near rims of the disk diameter (*id*) and outer diameter (*od*). The time-of-flight image showed that the transit time was smaller near the *id* and *od* of the disk and larger between *id* and *od*. It should be pointed out that since the width of the disk (94mm) was approximately 15 times that of the transducer diameter (6.35mm), these amplitude and transit time variations in the radial direction were not just due to edge effects, but were attributable to material property nonuniformity. The behavior of the ultrasonic transmission through the disk was consistent with changes in ultrasonic attenuation and velocity caused by density variation. Near the rims of the disk, the density was greater and the void content was lower; hence the attenuation was lower and the velocity was higher (Jeong and Hsu, 1995 and 1996). Between the *id* and *od*, the density was lower (higher void content); as a result, the attenuation was higher and the velocity was lower. These results were also consistent with the expectation of the pitch impregnation process. Because the pitch impregnation was most effective



**Fig. 5** Displayed are peak-to-peak amplitude and time-of-flight of through-transmission for the production C/C brake disk. Transmitter and receiver are 5MHz, 6.35mm diameter and no focus transducer. Scan areas were  $330 \times 330$ mm

along paths between the plies, the regions near the *id* and *od* should be densified to a greater degree than the region between the *id* and *od*. The images in Fig. 5 also showed a certain degree of circumferential nonuniformity. If one draws a dividing line along the 5 O'clock to 11 O'clock direction, then the left half of the disk seemed to be more densified than the right half of the disk.

### 3.2 Ultrasonic velocity measurements

Using the method illustrated in Fig. 3, the longitudinal wave velocity in the thickness direction was measured at 24 locations shown in Fig. 6. Locations adjacent to *id* and *od* are shown with solid diamonds and interior locations are shown with empty diamonds (the same convention used in Fig. 7). The measured velocities varied between  $1.63\text{mm}/\mu\text{s}$  and  $1.82\text{mm}/\mu\text{s}$ . A plot of the velocity versus position, as shown in Fig. 7, revealed a highly regular pattern, the velocities at every point between the *id* and *od* were lower than those at the adjacent *id* and *od*. The measurements were repeated three times and the data are shown in Fig. 7 as Run 1, Run 2 and Run 3. The velocity data measured with dry-coupling transducers provided strong evidence of

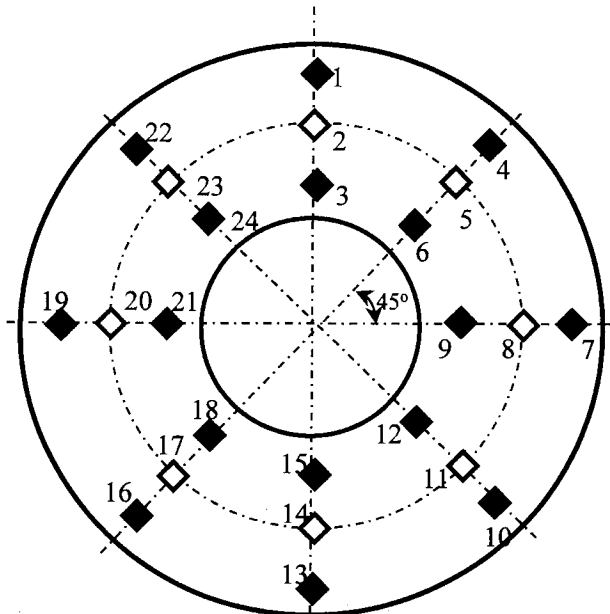
a consistent material property nonuniformity in the radial direction.

### 3.3 Destructive analysis

To verify that the above interpretation that the nonuniformities of the disk revealed by ultrasonic attenuation and velocity were caused by nonuniform impregnation and the resulting density variation, six small sample were cut out along the radius at the 3 O'clock direction. These samples, labeled No. 1 through No. 6 as shown in Fig. 1, were individually weighed and their densities determined. The results are shown in Table 1. The density was clearly higher near the *id* and *od*, and lower in the middle region. The longitudinal wave velocity in the thickness direction was measured for each of the six cut-out pieces and the results were correlated with the densities. Figure 8 shows a clear correlation between ultrasonic velocity and density. For regions of a high degree of densification, the porosity content was low, the density was high, and the ultrasonic velocity was high. These regions had a greater stiffness. Conversely, regions of poorer densification had lower density, lower velocity and lower stiffness.

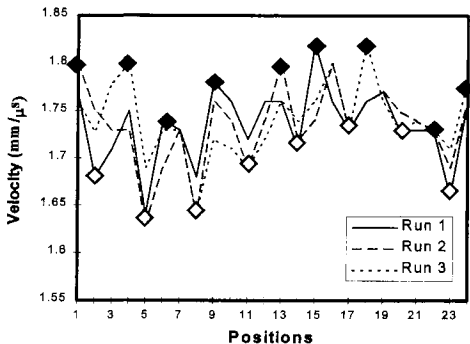
**Table 1** Thermal and mechanical properties used in finite-element analysis

Material	Young's modulus E(GPa)	Thermal expansion Co. $\alpha (\times 10^{-6}/^{\circ}\text{C})$	Poisson ratio $\mu$
PSZ	$E=45.06-0.0248T$ ; T in $^{\circ}\text{C}$	100 $^{\circ}\text{C}$ - 9.75 600 $^{\circ}\text{C}$ - 10.9 400 $^{\circ}\text{C}$ - 10.7 1000 $^{\circ}\text{C}$ - 10.7	0.23
NiCrAlY	$E=125.06-0.0149T$ ; T in $^{\circ}\text{C}$	100 $^{\circ}\text{C}$ - 12.5 600 $^{\circ}\text{C}$ - 15.7 400 $^{\circ}\text{C}$ - 14.3 1000 $^{\circ}\text{C}$ - 16.5	0.23
STS304	$E=201.44-0.1643T$ ; T in $^{\circ}\text{C}$	100 $^{\circ}\text{C}$ - 17.3 400 $^{\circ}\text{C}$ - 17.9 600 $^{\circ}\text{C}$ - 18.7 1000 $^{\circ}\text{C}$ - 20.6	0.29



- 1) Point measurements were made at 24 positions on C/C composite brake disks using 1MHz dry-coupling transducers.
- 2) Filled diamond points are near exposed edges at the inner and outer diameters, and open diamond points are away from exposed edges.

**Fig. 6** Velocity measurement positions using dry-coupling transducer



**Fig. 7** Relationship between velocities and measurement positions

### 3.4 Detection of near-surface anomaly

Through-transmission ultrasonic scans sampled the integrated material property over the entire thickness; they were not particularly sensitive to subtle anomalies near the surface. To detect minor material property variation near the surface, one could make use of the small amplitude variation of the front surface reflection (Hsu, Haghes, and Patton, 1993). To examine the surface of the C/C brake disk, a 25 MHz, 6.35mm diameter, unfocused immersion transducer was used to generate a C-scan image of the front

surface echo amplitude. The resulting image, as shown in Fig. 9, revealed four circular rings equally spaced on the disk. Displayed are peak-peak amplitude of the front surface echo. These features were caused by the placement of "spacers" between adjacent C/C disks when a stack of disks was run through the final chemical vapor infiltration process. The spacers unavoidably impeded the infiltration and left a slightly less densified footprint underneath them. It was in fact confirmed that, for the disk in Fig. 9, the spacers used were tubular instead of solid cylinders, which explained the ring-shaped features observed.

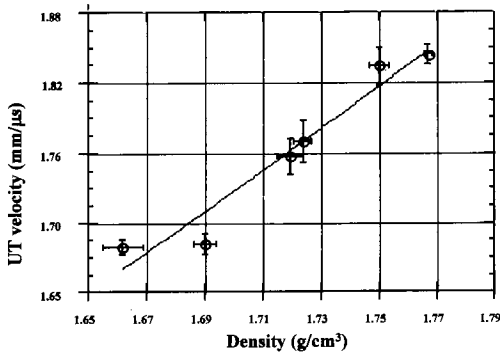


Fig. 8 Relationship between density and velocity for the six cut-out pieces

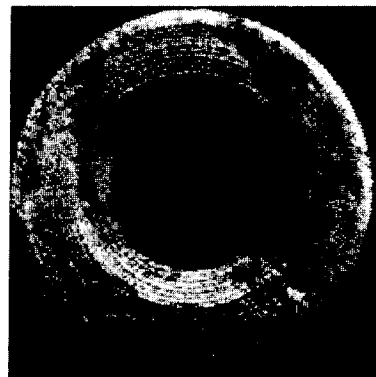
3.5 In-plane analysis from six cut-out pieces

Figure 10 shows the amplitude and time-of-flight images of six cut-out pieces, which show considerable variation in the thickness (vertical) direction. Figures 10 (a) and (b) indicate radial and circumferential direction images respectively and numbering order of Fig. 10 indicates the six cut-out piece order as shown in Fig. 1, selected 3 O'clock direction of the disk in Fig. 5. It has been found that the transmitted amplitude of UT results is higher and time-of-flight is longer on both radial and circumferential directions. The reverse phenomena appear if compared to Fig. 5. Figure 11 shows optical micrograph of one of six cut-out pieces, which consisted of mainly chopped fibers and matrix at a lower half region of No. 1 cut-out piece. Figure 12 represents schematic stacking sequences of a C/C brake disk, with which is not symmetrically composed. The stacking sequences were reconstructed based on optical micrograph (Olympus, BH2). Upper half had more cloth than chopped fibers; whereas lower half had more chopped fiber layups than cloth layups.

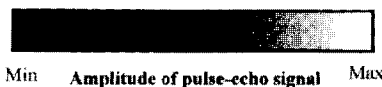
Figure 13 shows ultrasonic velocity in the radial and circumferential direction using the six cut-out pieces. Higher velocity on the upper half region appears lower than lower half on both radial and circumferential direction images



Transducer : 10MHz, 6.35mm dia. and no focus



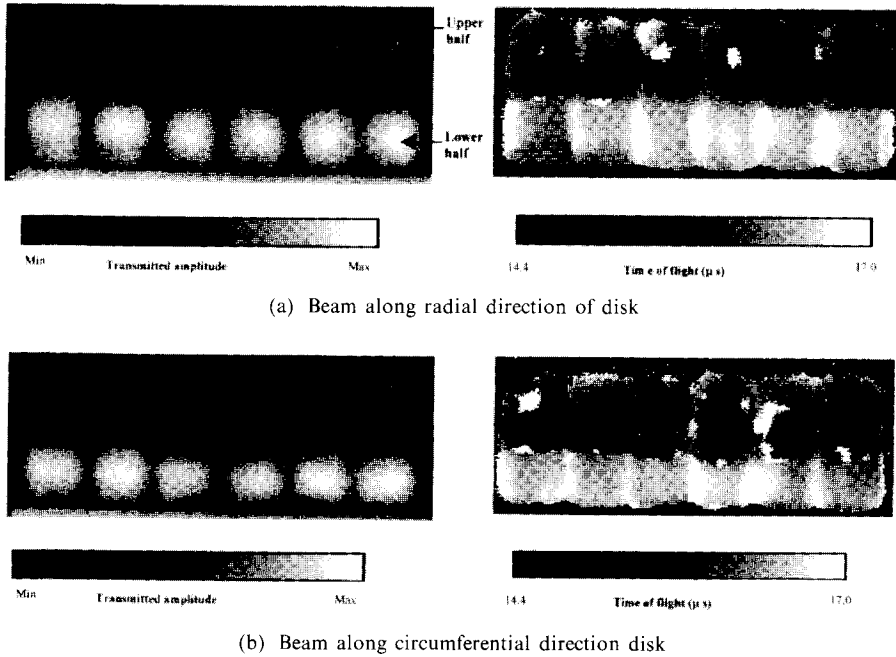
Transducer : 25MHz, 6.35mm dia. and no focus



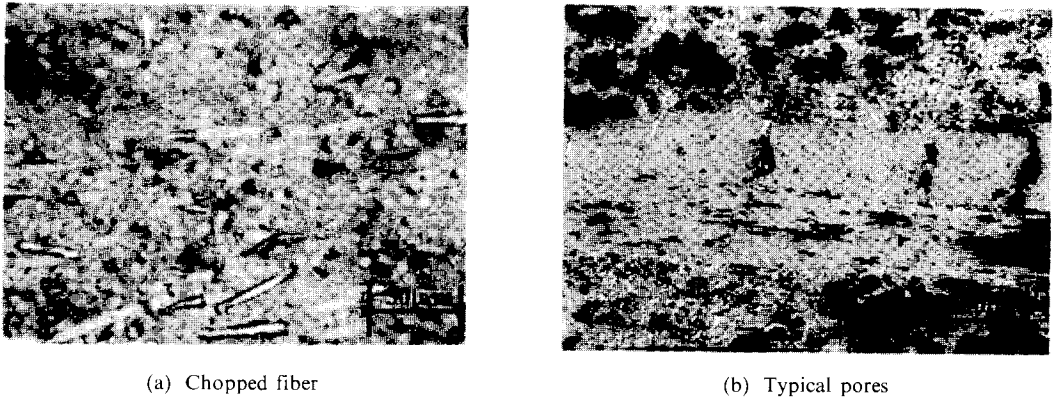
\* Displayed is peak-peak amplitude of front echo. Scan area is 330 x 330 mm.

Fig. 9 Ultrasonic scan image shows four anomalous regions on the disk





**Fig. 10** Displayed are peak-to-peak amplitude and time-of-flight of through-transmission signal for the six cut-out pieces. Transmitter and receiver are 5MHz, 6.35mm diameter



**Fig. 11** Optical micrographs of one of the cut-out pieces showing chopped fibers and matrix

respectively. Velocity of radial and circumferential direction appears approximately three times as great as that of thickness direction. Lower half had more chopped fiber and corresponded to lower velocity and lower attenuation. Upper half had more fabric and corresponded to higher velocity and higher attenuation. Therefore, the in-plane velocities in the radial and circumferential direction are essentially independent of density unlike the velocity in the thickness direction.

However, the upper half and lower half had clearly different velocities due to their different contents of chopped fiber and fabric.

#### 4. Conclusions

The manufacturing process for C/C composites is rather complex and cost a lot compared with conventional composites due to the multiple steps. Nondestructive test techniques have been

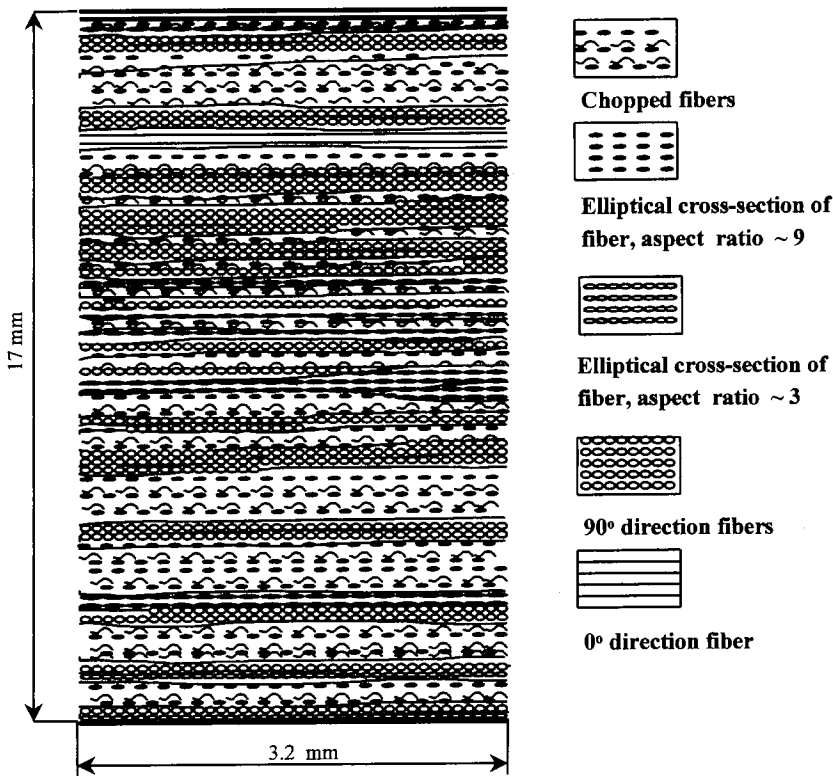


Fig. 12 Schematic stacking sequences of a C/C brake disk reconstructed from optical micrographs

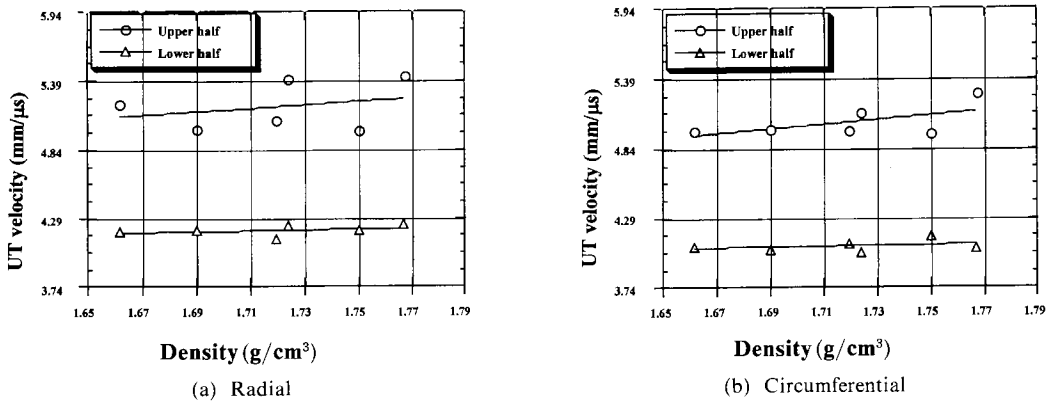


Fig. 13 Relationship between density and velocity on the six cut-out pieces in the radial and circumferential directions

conducted with C/C composite brake disks.

In this work, several ultrasonic techniques were able to yield useful information about material property nonuniformity of a carbon/carbon brake disk using both immersion scanning and dry-coupled contact method. Through-transmission C-scan images provided visual, qualitative

pictures for radial and circumferential nonuniformity. Pulse-overlap velocity measurements using dry-coupled transducers gave quantitative data for correlation with C-scan images and with material density variation. A consistent pattern of variation along the radial direction existed for ultrasonic attenuation and velocity. This varia-

tion was attributed to the density variation as confirmed by destructive analysis. The higher density occurred near the inner and outer diameters of the disk, where the ultrasonic attenuation was lower and the velocity was higher which was caused by the more efficient densification by pitch impregnation near the *id* and *od* and by the less efficient densification away from the exposed edges of the disk. Ultrasonic scans of the surface were also able to reveal subtle, near-surface material property anomalies attributable to the manufacture process. Also, ultrasonic velocities in the in-plane directions (radial and circumferential) seemed to be affected more by the relative contents of fabric and chopped fiber, and less by the void content.

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