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**Measurement of Elastic Moduli of Porous SiC/SiC Ceramic Matrix
Composites Using Ultrasonics**

by

Vikram Saini

A Report Submitted to the
Graduate Faculty in Partial Fulfillment of the
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Measurement of Elastic Moduli of Porous SiC/SiC Ceramic Matrix Composites Using Ultrasonics

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ABSTRACT

Forced Chemical Vapor Infiltration (FCVI) method was used in fabricating the NicalonTM/Silicon Carbide composites. Both through-thickness and In-plane (fiber fabric plane) moduli were determined using ultrasonic techniques. The through-thickness moduli were found to be much less than the in-plane moduli. Both in-plane and through-thickness moduli were significantly decreased with an increased porosity. The experimental results for the moduli were found to be in good agreement with a theoretical model.

Introduction

As high temperature requirements for structural materials become more and more stringent, toughened ceramic matrix composites (CMC) consisting of fibers or whiskers dispersed in a ceramic matrix are being developed as a potential solution. These CMCs are required to exhibit high temperature strength, improved fracture toughness and high hardness. Other needs are for corrosion- or oxidation resistant wear surfaces and surfaces which are thermal insulators with high temperature stability. In some applications, new bulk high temperature materials are also in demand, which overcome the traditional brittleness of refractory materials. Ceramic composites appear to meet the needs, with ceramic fibers infiltrated with a ceramic matrix. High strength of the fibers enables the transfer of high load from the matrix to the fibers. Ceramic matrix composites have a high fracture toughness because energy is absorbed as the fibers pull out of the matrix, causing crack deflection or blunting. Many conventional processes for the fabrication of ceramics tend to mechanically, thermally or chemically damage the fibers during processing. Until just a few years ago, vapor-phase synthesis was rarely considered for the fabrication of structural materials. Now, however, vapor-phase infiltration has become the fabrication method of choice with a class of techniques termed Chemical Vapor Infiltration (CVI).

There are five general classes of CVI techniques shown in Fig. 1: (1) isothermal (2) thermal gradient (3) isothermal-forced flow (4) thermal gradient-forced flow and (5) pulsed flow. The isothermal-forced flow process, which uses forced convection, suffers from density gradients and loss of permeability at the entrance surface. The thermal gradient forced-flow- technique developed at Oak Ridge National Laboratory (ORNL) has been used in fabricating the CMCs for this experiment. Forced Chemical Vapor Infiltration (FCVI) technique overcomes the problems of slow diffusion and restricted permeability and is capable of producing thick-walled, simple-shaped components in times on the order of hours, significantly reducing the production costs, whereas, the processing for the first three CVI techniques has been extremely long on the order of weeks. The FCVI process is self-optimizing, which has allowed for much shorter infiltration times and less difficulty in obtaining uniform infiltration.

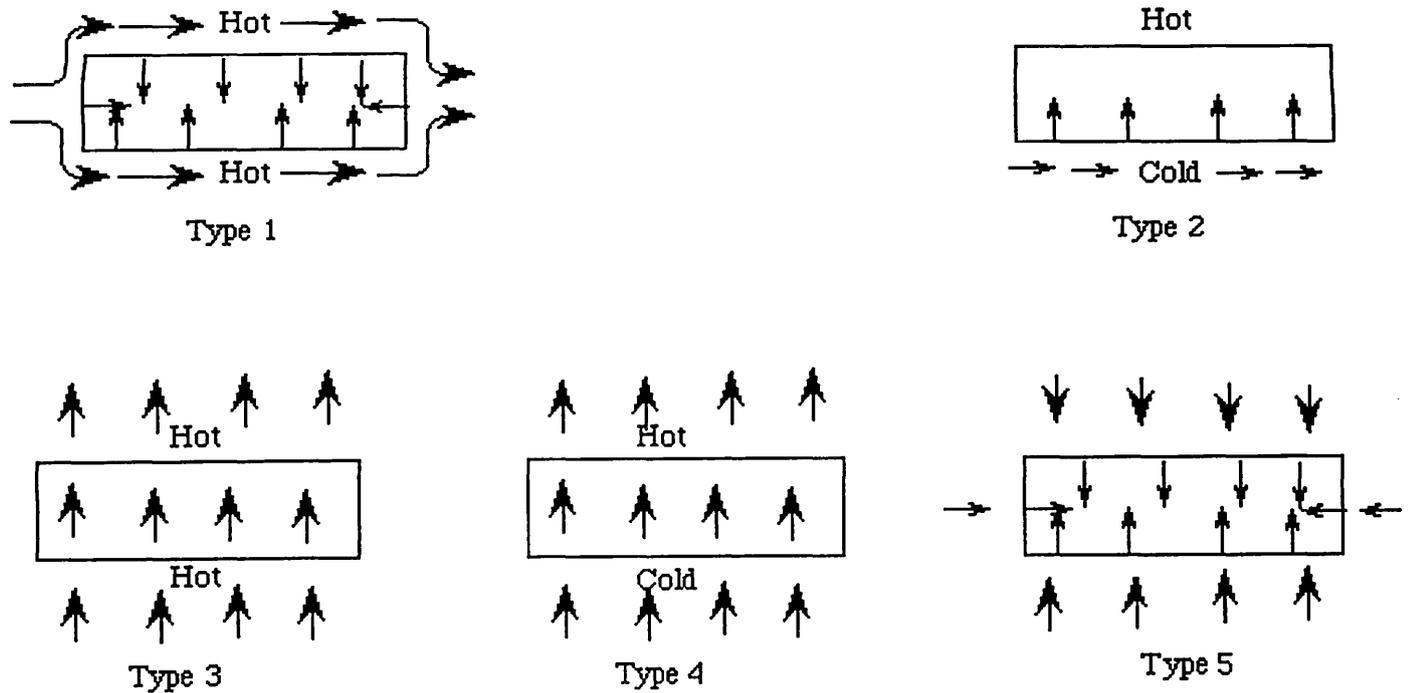


Fig. 1. The five general classes of CVD techniques.

Upto the present time, relatively little work has been performed on the mechanical behaviour of woven fabric reinforcement CMCs. There is little theoretical modeling work to predict the moduli of woven fabric composites in open literature. In this investigation, moduli of Nicalon fiber fabric reinforced SiC composites were measured using an ultrasonic technique. These results are compared with model predictions obtained by University of Tennessee.

Material

Fibrous Preforms and Matrix Materials

Several oxide, carbide and nitride fibers that offer a variety of properties to the material designed have become available in recent years. The most commonly used SiC fiber, Nicalon, a polymer-derived microcrystalline/amorphous material is used with silicon carbide matrix in fabricating the CMCs for this investigation. Nicalon contains the chemical composition (in weight%) of 59% Si, 31% C and 10% O₂. Preform geometries can be tailored to the application to maximize strength and toughness in the

direction of maximum stress. A preform was made by stacking Nicalon fiber cloth plies for reinforcements within the plane of cloth. To control the mechanical properties of Nicalon-SiC composite a thin pyrolytic carbon layer is deposited on fibrous preforms before densification to provide a controlled and uniform interface with the matrix. The coating prevents chemical damage of the fibers during processing and prevents weakening of the fiber matrix interface, enhancing fiber debonding and slip. Thus, the coating results in an increase in the toughness and the ultimate strength of the composite material.

Method of Fabrication

In CVI, gaseous reactants infiltrate a porous (typically fibrous) preform held at an elevated temperature depositing matrix material on the substrate structure via a standard chemical vapor deposition (CVD) reaction. This process simultaneously utilizes thermal and pressure gradients to reduce the infiltration time from weeks to hours. The CVD coating grows with continued deposition to form the composite matrix. A schematic of the process is shown in Fig. 2. Fibrous preforms are retained within a graphite holder that contacts a water-cooled metal gas distributor, thus cooling the bottom and side surfaces of the substrate. A steep temperature gradient across the preform is created by exposing the top of the fibrous preform to the hot zone of the furnace. The reactant gases, methyltrichlorosilane (MTS) and Hydrogen, are forced under pressure into the cooled side of the fibrous preform where they initially do not react because of the low temperature. The gases continue from the cooled portion of the preform into the hot portion, where they decompose and deposit on and around the fibers to form the Silicon Carbide Matrix. Deposition of matrix material within the hot region of the preform increases the density and thermal conductivity of the preform, so the deposition zone moves progressively from the hotter regions towards the cooler region or in other words from the top of the preform toward the bottom. The process continues until reduced permeability of the densified composite prevents sufficient flow of reactant gases into the preform. The composites thus fabricated had a nominal fiber volume fraction of 0.4.

The FCVI process is well suited for the fabrication of thick-walled composites of relatively simple

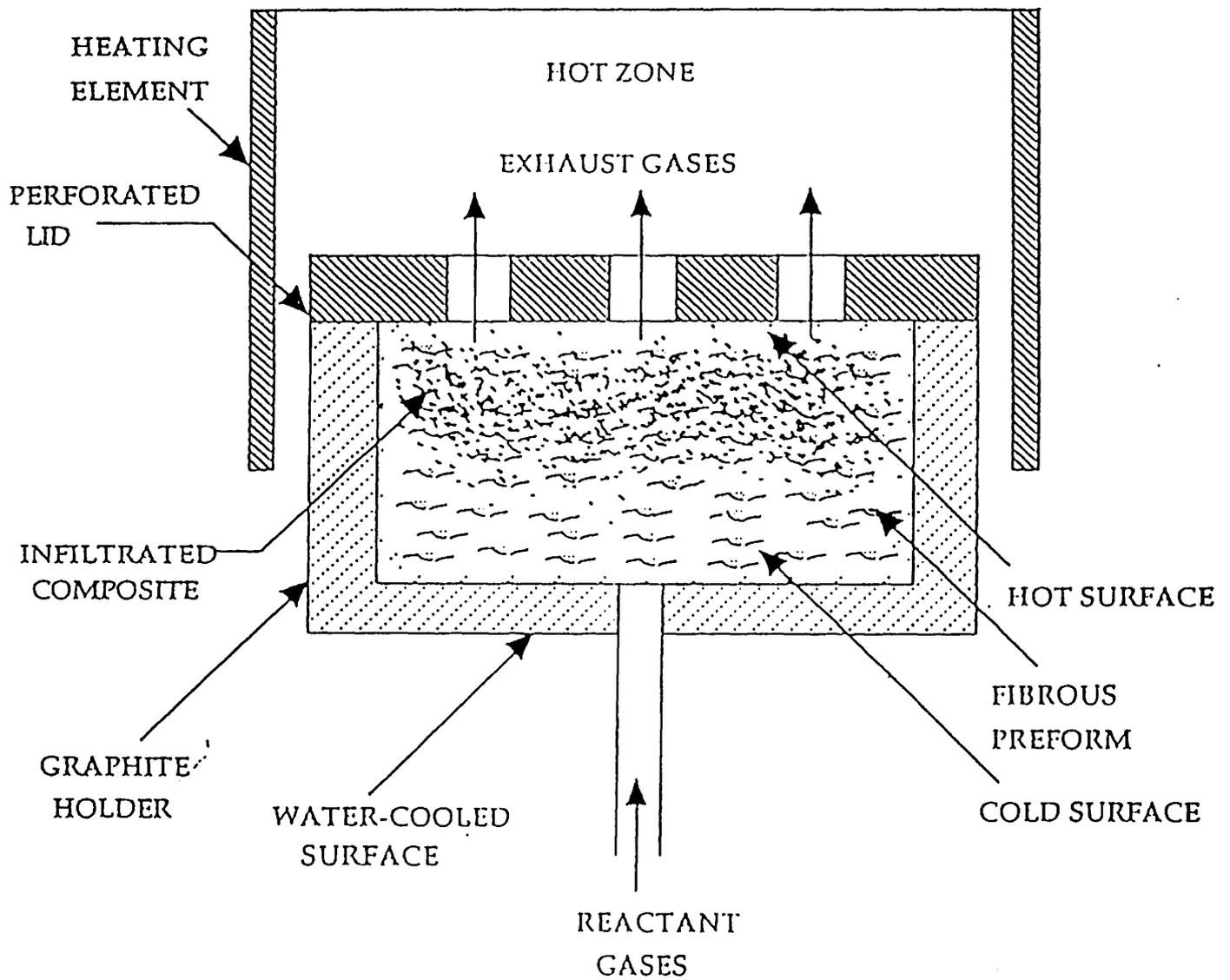
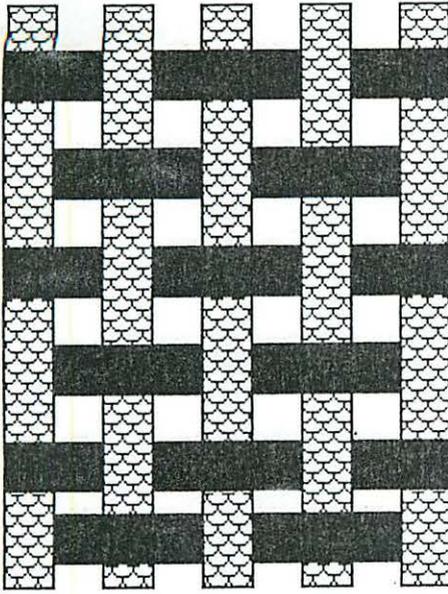


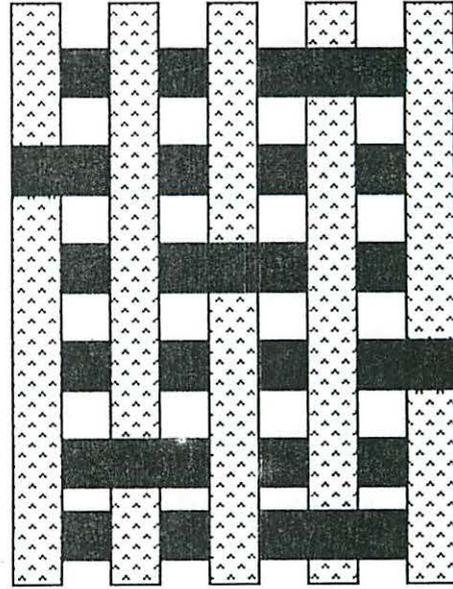
Figure 2. Schematic of the Forced Chemical Vapor Infiltration (FCVI) Process to Fabricate Nicalon/SiC Composites

shapes. Several advantages are inherent in this process, the primary one being increased efficiency and reduced infiltration times. Second, the fabrication of preforms is simplified because fibrous materials are held in place by graphite holder instead of requiring careful processing with binders that must be gently removed. Third, moderate pressures can be applied to the graphite holder to compress the preform and increase the fiber loading of the composites. Fourth, composites with greatly increased thicknesses can be efficiently fabricated.

The primary objectives of CVI are to maximize the rate of matrix deposition and minimize density gradients. Unfortunately, there is an inherent competition between the deposition reaction and the mass transport of the gaseous species. Higher infiltrated densities are difficult to obtain because the permeability of the preforms is quite anisotropic: The permeability along the axis of the fibers is much greater than the permeability perpendicular to the fibers. Therefore, reactant gases fail to effectively infiltrate into the center of the preform and instead permeate along the length of the fibers until they deposit near the circumference of the composite. Uniform deposition can be obtained throughout the composite by infiltrating cloth preforms, but large voids are present. Flaws are created between tows in the weave and are frequently quite large because the voids in numerous layers align over one another. More uniform preforms with much finer porosity must be produced in order to infiltrate to higher densities and thus obtain higher strengths and improved strain behaviour. The fiber fabrics are typically categorized into plain and satin weaves, as shown in Fig. 3. In this work, plain weave was used to fabricate the composite. The fiber fabric was rotated in 30° increments (i.e., fiber orientations in each ply were [0/90], [30/120] and [60/150]). Approximately forty plies were present per centimeter thickness. Each sample contained 15 layers of woven Nicalon fiber cloth.



(a)



(b)

Fig. 3. Fiber Fabric Architecture: a) Plain Weave b) Satin Weave.

Although cloth preforms appear to be equally permeable in all directions, the samples fabricated using the FCVI process seem to have some porosity. Density of each sample was determined from its weight and its volume based on the outer dimension of the sample. It is assumed that the void free density for SiC/SiC samples is 2.97g/cc. This is based on a density of 2.6 g/cc for the Nicalon fibers, a density of 3.21 g/cc for the SiC matrix and a fiber volume fraction of 0.4. The effect of the pyrolytic graphite coating, about 0.3 μm in thickness, is ignored in the void content estimation. The void % in the samples is calculated using Eq. (1).

$$\text{Volume percent of Void} = \left(1 - \frac{\text{Measured density}}{\text{Void free density}} \right) 100 \quad (1)$$

The samples used for the experiment had a porosity ranged from 11% to 32% by volume. The goal of the experiment was to find out the effect of porosity on the elastic properties of Nicalon/SiC Ceramic Matrix Composites.

Experimental Procedure

Moduli measurements were performed using an ultrasonic technique. Longitudinal wave, dry coupling transducers were used in the determination of the longitudinal ultrasonic velocities in the thickness direction and in-plane directions of the samples. The longitudinal modulus of elasticity C , or the elastic stiffness constant for the direction of propagation is obtained using the simple relationship of Eq. (2) given below.

$$C = \rho V_L^2 \quad (2)$$

where, ρ = density of the sample and

V_L = longitudinal velocity

Longitudinal wave velocity in a material could be measured using one of the following ultrasonic methods: (a) Dry contact, (b) Water Immersion, or (c) Contact using liquid or gel couplant. Since the samples used in this study are highly porous, the use of liquid or gel couplant will change the value of the modulus and cause contamination of the samples. To avoid these problems, ultrasonic measurements were performed using dry coupling longitudinal transducers. Dry contact transducers have a thin elastomer face-sheet and couple the ultrasonic propagation by pressure, without the aid of liquid or gel couplant. The transducers have a crystal size of 0.75" and a center frequency of 0.5 MHz.

The experimental setup for measuring the ultrasonic time of flight through a sample is shown in Figure 4. The transmission setup requires a pair of transducers; one of which serves as a transmitter while the other serves as a receiver. In order to achieve good accuracy in time of flight (TOF) measurements, a

pulse-echo overlap method was used to measure the TOF through the sample. In this method, TOF *through the sample* is the time difference between a reference signal and the sample signal. The diagram on the left shows the arrangement for acquiring the reference signal. A piece of fused quartz is placed between the transducers as the reference material. A thin sheet of rubber is also placed between the transducers because it is needed in acquiring the sample signal. A digital oscilloscope is used to capture and display the through-transmitted longitudinal wave echo. The diagram on the right shows the arrangement for acquiring the sample signal. The rubber sheet provides a coupling between the sample and the reference piece. The through-transmitted echo is again acquired by the oscilloscope. It

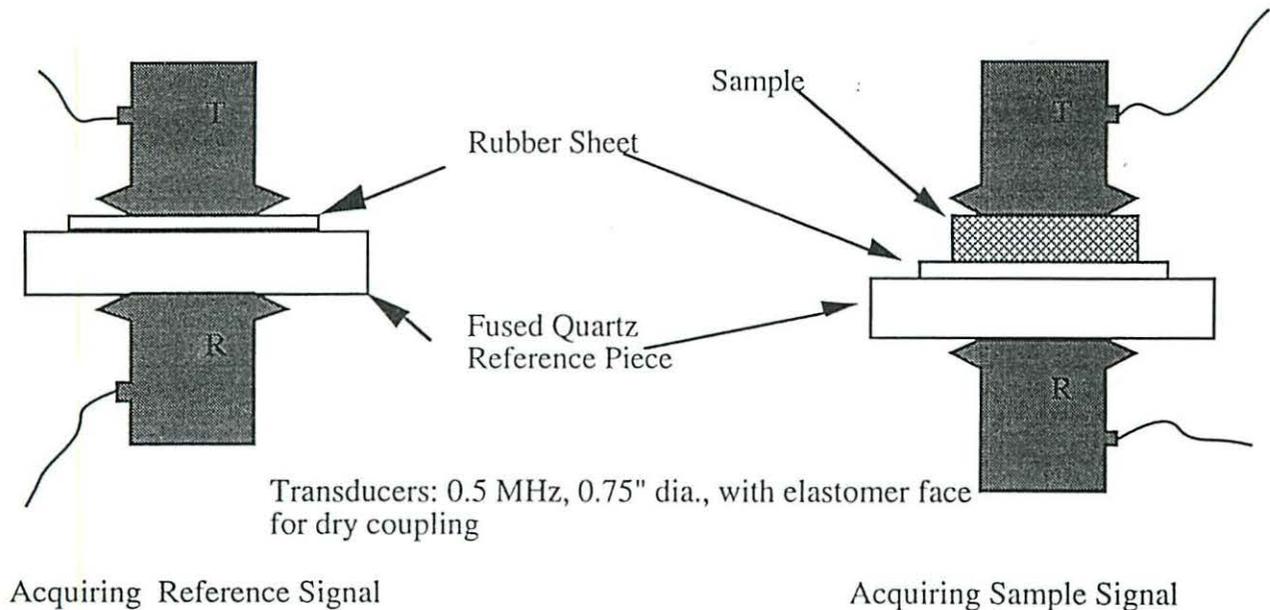


Fig. 4. Experimental method for acquiring the reference and sample signals.

is clear from a comparison of the two diagrams that the difference in the TOF of the two signals is simply the transit time through the thickness of the sample. On a digital oscilloscope (e.g. LeCroy 9000 series), this time difference can be accurately determined by shifting one signal to overlap with the other. The amount of time shift is the TOF through the sample. Because of frequency dependent attenuation and dispersion in the porous sample, the reference signal and the sample signal may be somewhat different in waveform. In such cases, the pulse-echo overlap is based mainly on the first few peaks of the radio frequency (RF) pulse and differences in the trailing signal are ignored.

Velocity measurements were made for two orientations: (a) through the thickness of the square-plate samples and (b) in the plane of the square plates. Figure 4 shows the setup for a through-thickness velocity measurement since wave travels through the thickness when sample is oriented in the manner depicted. In the in-plane velocity measurement, the sample is placed on its edge i.e. (standing up) between the transducers, allowing the wave to travel in the in-plane direction. The samples were placed in two orthogonal in-plane directions (0° and 90°) for in-plane velocity measurements. Experiments were done for both the in-plane directions and the results are shown in Fig. 5. It is seen that longitudinal velocity is approximately the same for the two in-plane directions. This is to be expected because each layer of cloth contains 0° and 90° fibers; the laminate therefore possesses a 90° rotational symmetry.

In the configuration for in-plane velocity measurements, the transducer diameter is considerably greater than the sample thickness. The edge effect that may affect the velocity measurements is therefore a concern. Due to the lack of SiC/SiC samples of different thickness, graphite epoxy and fused quartz

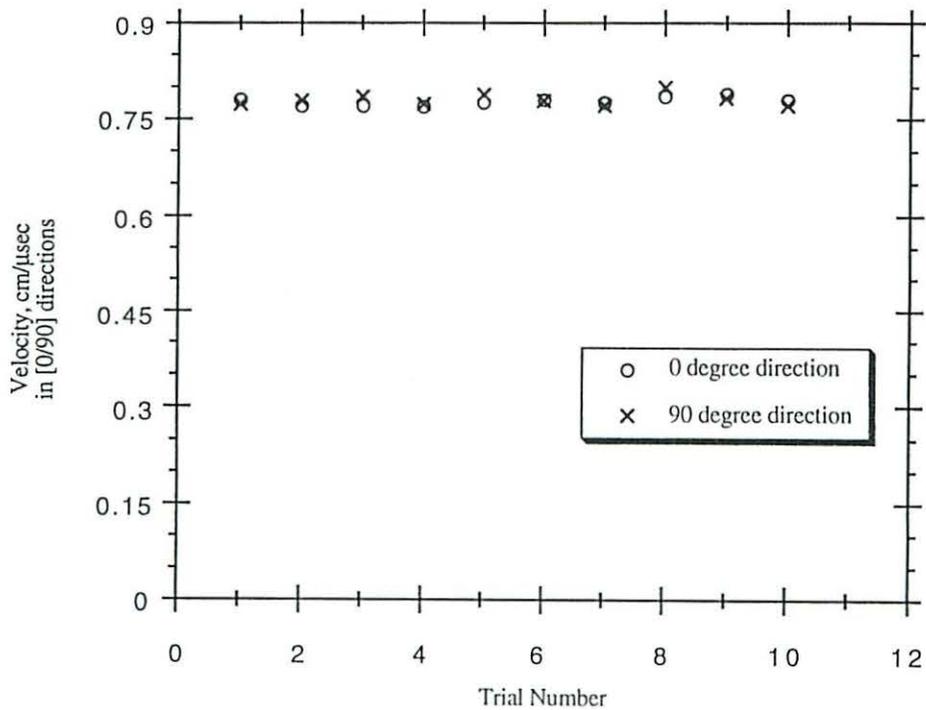


Fig. 5. A plot between longitudinal velocity in two in-plane orthogonal directions and number of trials.

pieces of various thickness were used to assess the edge effect. Graphite epoxy samples were used *because, like SiC/SiC samples, they are also laminated.* Fused quartz was used because the speed of sound is well known in fused quartz and because its thickness was close to that of the SiC/SiC samples.

It was found that sample thickness in this experiment only affected the longitudinal velocity by a few percent. The edge effects in the in-plane velocity measurements are therefore ignored in this preliminary study.

Results

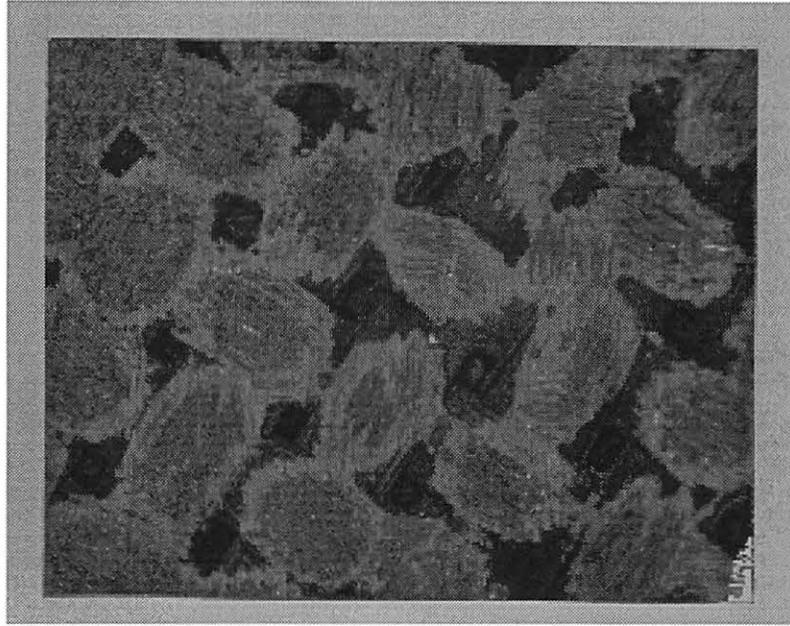
Microscopy

Microscopic examination of the composite was carried out to examine the type and distribution of porosity in the samples. Planar and cross-sectional views of a composite specimen are shown in Fig. 6. It could be seen from Fig. 6 that there were two major types of porosity in the Nicalon/SiC composite, viz. porosity at the fiber tow intersection [Fig. 6(a)] and interlaminar porosity [Fig. 6(b)]. The extent of interlaminar porosity was found to be greater than the porosity present at the fiber tow intersection. The composite was highly anisotropic and hence, it could be expected that the mechanical properties of the Nicalon/SiC composites will vary significantly along the in-plane and through-thickness directions.

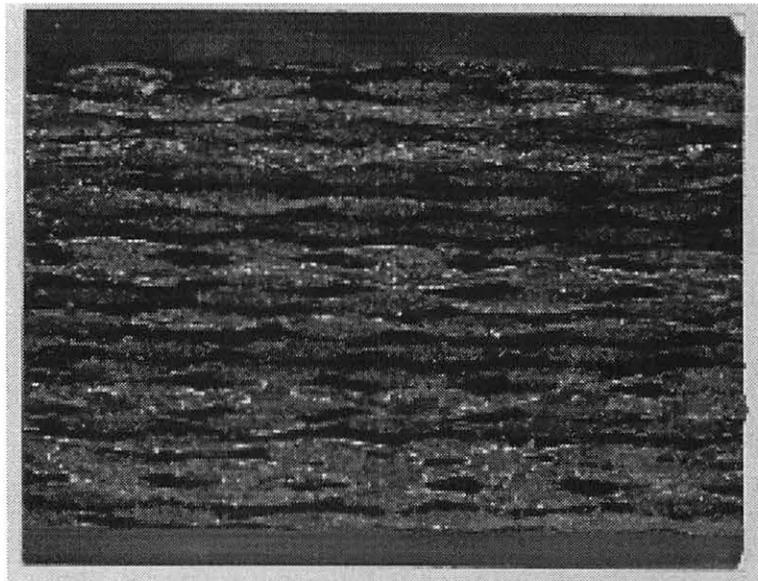
The density (as measured from the specimen weight and dimensions) and % porosity of the specimens are listed in Table 1. Note that the % porosity in the sample is given by Eq. (1). The density of the samples varied from 2.02 g/cc to 2.63 g/cc and the % porosity from 11.5 to 31.6. The theoretical density of the composite is 2.96 g/cc, which is based on the density of 2.6 g/cc for the Nicalon fibers and 3.21 g/cc for the SiC matrix, and a fiber volume fraction of 0.4. The variation of the porosity allowed us to evaluate the effect of porosity on the moduli of the composites.

Ultrasonic Measurements

The longitudinal moduli of the composites were determined in both in-plane and through-thickness



(a) Planar View



(b) Cross-sectional View

Fig. 6. Planar and Cross-sectional Views of Nicalon/SiC Composites

Sample No.	Vel.(cm/ μ sec),through	Vel.(cm/ μ sec),in-plane	Density (g/cc)	Porosity(%)	*Modulus1, GPa	#Modulus2, GPa
1	0.471	0.987	2.55	14.141	56.569	248.41
2	0.477	0.990	2.45	17.609	55.676	239.83
3	0.465	0.994	2.45	17.340	53.083	242.56
4	0.364	0.927	2.30	22.559	30.474	197.65
5	0.377	0.927	2.33	21.549	33.116	200.22
6	0.378	0.936	2.35	20.875	33.578	205.88
7	0.220	0.814	2.07	30.303	10.019	137.16
8	0.219	0.799	2.03	31.650	9.7361	129.60
9	0.227	0.812	2.03	31.650	10.460	133.85
10	0.512	0.972	2.42	18.519	63.439	228.64
11	0.442	0.957	2.38	19.865	46.497	217.97
12	0.552	0.972	2.40	19.192	73.129	226.75
13	0.167	0.877	2.02	31.987	5.6336	155.36
14	0.195	0.897	2.06	30.640	7.8332	165.75
15	0.179	0.873	2.09	29.630	6.6966	159.28
16	0.725	0.967	2.63	11.448	138.24	245.93
17	0.685	0.950	2.62	11.650	123.12	236.82
18	0.653	0.956	2.60	12.559	110.74	237.35
19	0.629	0.949	2.26	23.771	89.573	203.90
20	0.525	0.951	2.34	21.380	64.358	211.18

* Modulus1 = Longitudinal Modulus in through-thickness direction.

#Modulus1 = Longitudinal Modulus in in-plane direction.

Table 1. Experimental Results using Ultrasonics.

orientations. The results are summarized in Table 1. The ultrasonic velocities and longitudinal moduli values are plotted against % porosity in Figs. 7 and 8 respectively. It should be pointed out that the longitudinal modulus measured in this experiment is not Young's Modulus. Young's Modulus, E is a function of shear velocity, longitudinal velocity and the density of the material. In this work, to date, only the longitudinal velocities are measured. Each data point in Figs. 9 and 10 represents an average of three repetitive measurements. The repeatability of the measurements, as indicated by the vertical

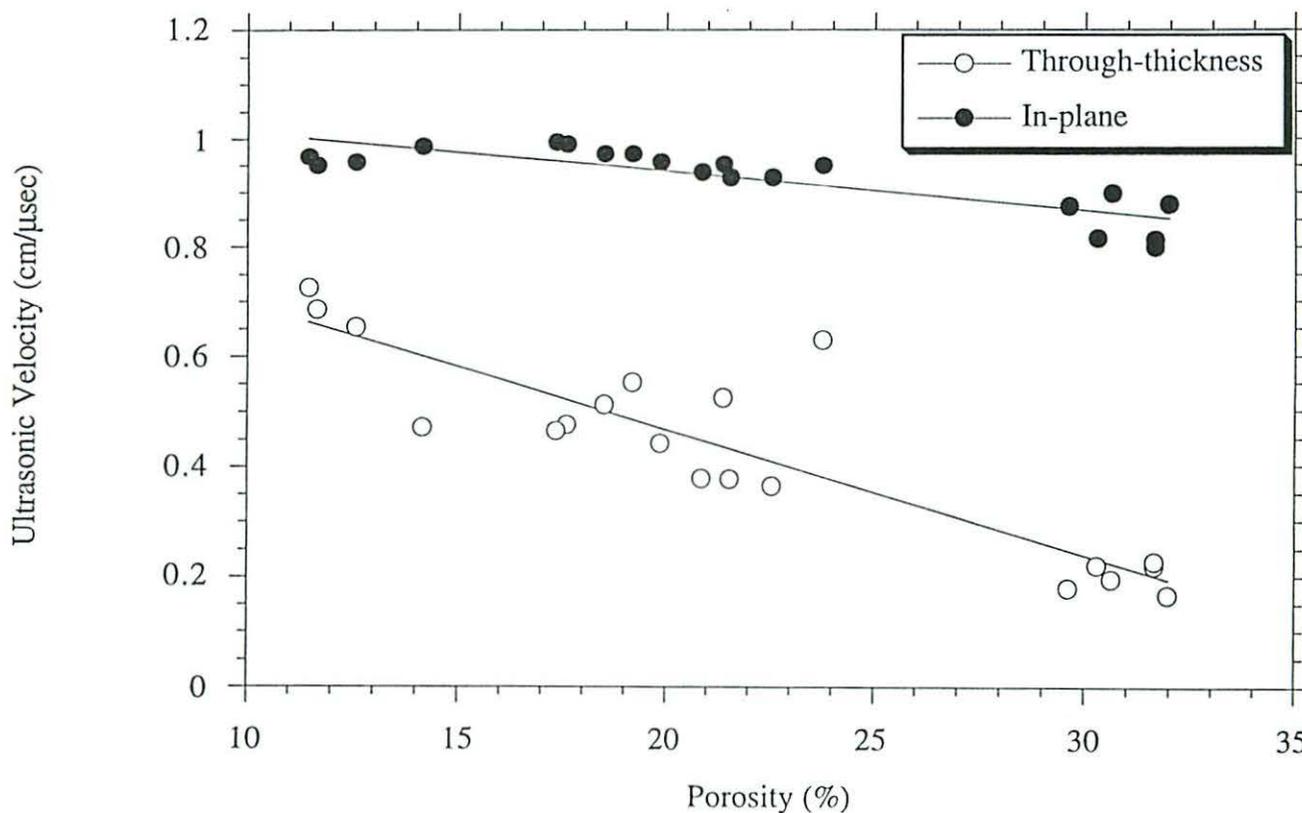


Fig. 7. Longitudinal Ultrasonic Velocity Vs % Porosity in Nicalon/SiC Composites

error bars on some data points, is in fact reasonable. The scattering in the data set as a whole is believed to be attributable to several reasons. The measurement results using the dry coupling transducers depend somewhat on the applied pressure. The applied pressure may not have been consistent from

sample to sample. Specimens with the same void content (volume fraction) may still have different void size and shape. This could degrade the correlation between the measured modulus and the measured modulus and the void content. Finally, nonuniform distribution of voids in the sample can also affect the modulus results. Nonuniform void content across the thickness has actually been observed on the cross section of some samples. Considering the complex nature of the porosity and the host structure, the correlation between the modulus and void content shown in Figs. 9 and 10 can be regarded as a useful relationship.

The SiC/SiC ceramic matrix composite samples are fabricated from a stack of woven Nicalon cloth; there are no fiber reinforcements in the thickness direction. The elastic stiffness in the plane of the laminate is therefore expected to be much greater than that in the thickness direction. It could be seen

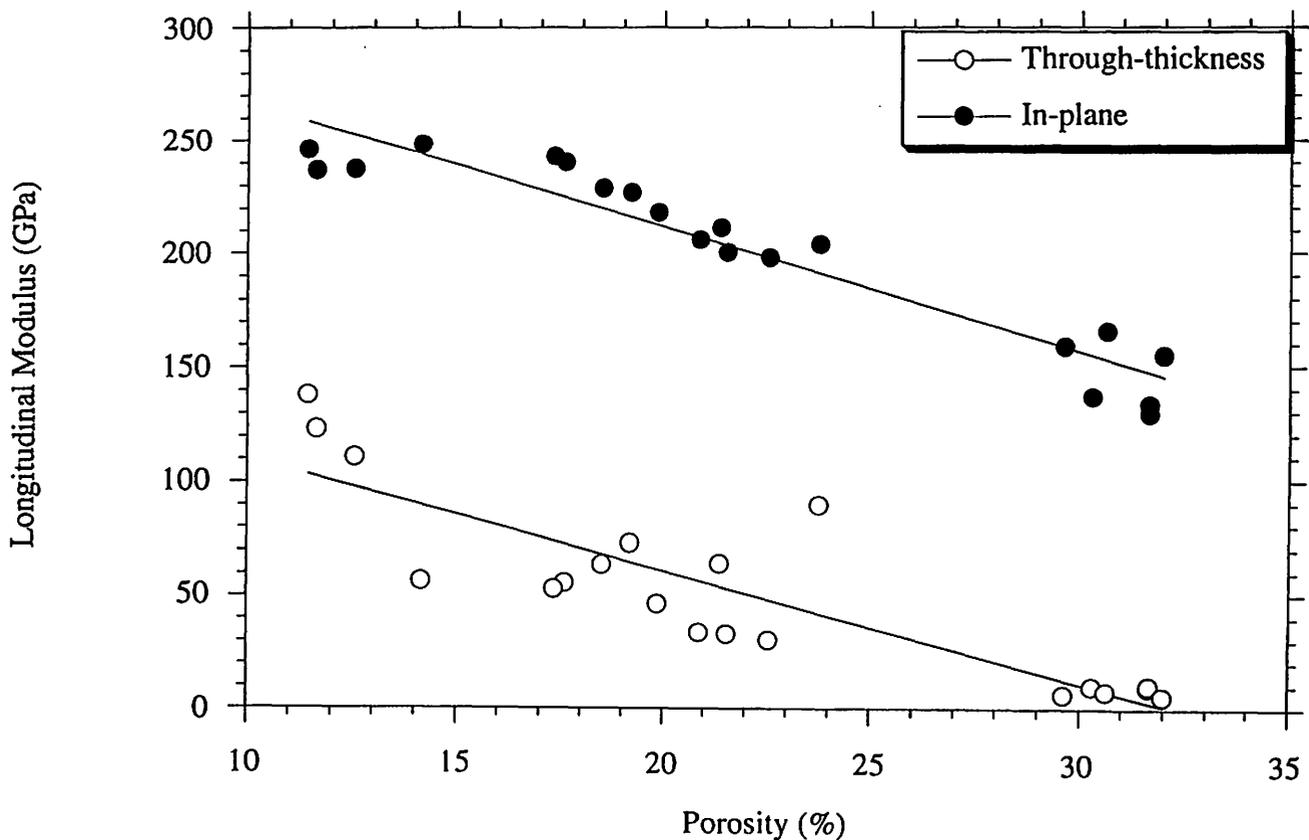


Fig. 8. Longitudinal Moduli Vs % Porosity in Nicalon/SiC Composites

by the experiment results in Figs. 7 and 8 that the ultrasonic velocities (0.7999 cm/ μ sec to 0.994 cm/ μ sec in the in-plane direction were greater than the ultrasonic velocities (0.167 cm/ μ sec to 0.725 cm/ μ sec) in the through-thickness direction. Consequently, the in-plane longitudinal moduli (133.85 GPa to 248.41 GPa) were greater than the through-thickness longitudinal moduli (6.69 GPa to 138.24 GPa), as could be expected from the fabric layup.

It can be seen that the longitudinal modulus in both directions decreases as porosity increases; however, porosity has a greater effect in reducing the longitudinal moduli in the through-thickness direction than in the in-plane direction. This behaviour can also be understood based on the morphology of the voids and the reinforcement direction. Microscopic examination of the cross-section of samples shows that the larger voids are mostly interlaminar and are flatted out to lie parallel to the plane. Because of their shape and orientation, such interlaminar voids are particularly effective in decreasing the velocity of longitudinal waves traveling along the thickness direction. For the in-plane modulus, on

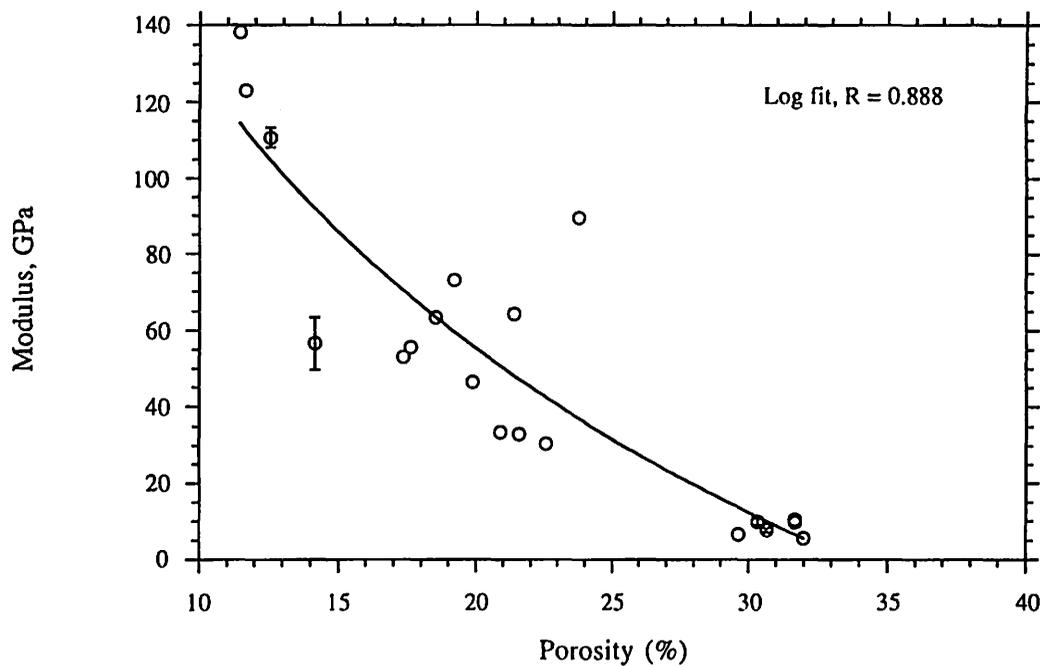


Fig. 9. Measured Longitudinal Modulus in the through-thickness direction as a function of porosity volume percent.

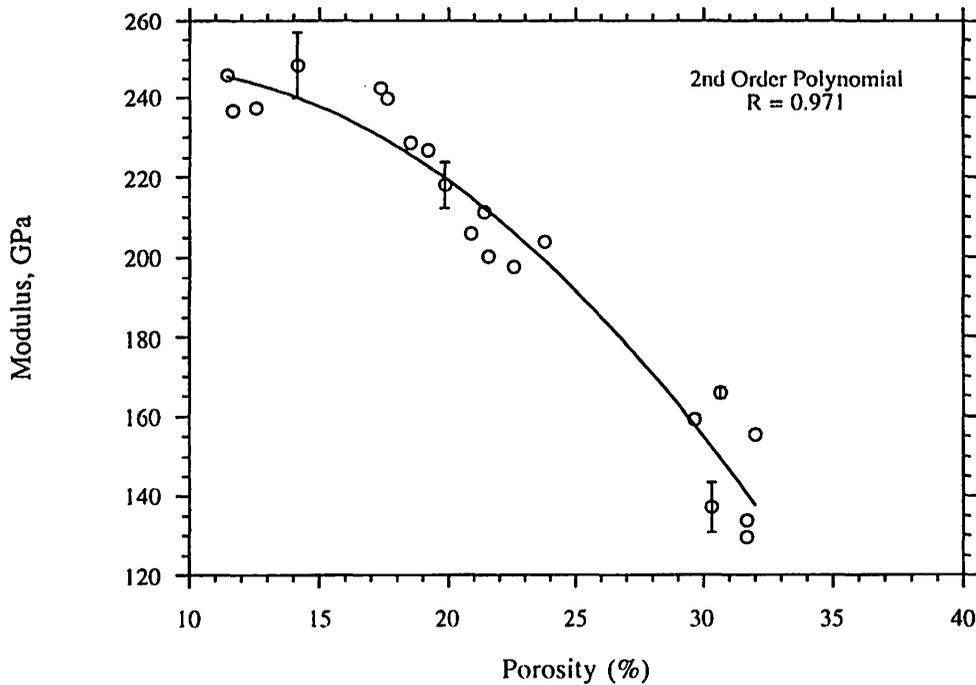


Fig. 10. Measured Longitudinal Modulus in the in-plane direction as a function of porosity volume percent.

the other hand, the continuous fibers provide most of the stiffness, and the effects of voids are not nearly as great as in the thickness direction. It should be noted that the ultrasonic measurements yielded comparable longitudinal moduli along the two orthogonal fiber fabric in-plane directions.

While taking the in-plane velocity measurements, it was noted that the transducer diameter was considerably greater than the thickness of the samples. In general, longitudinal velocity in thin rods is less than the longitudinal velocity in bulk material. Therefore the edge effect that may affect the velocity measurement was a concern. Due to the lack of SiC/SiC samples of different thickness, graphite epoxy and fused quartz pieces of various thickness were used to assess the edge effect. For the given thickness of the SiC/SiC samples, there was no significant edge effect present in this experiment.

Theoretical Model

A micromechanics model based on a periodic structure is developed at University of Tennessee^{1,3} to estimate the effect of porosity on the elastic properties of the woven fabric composites. As shown in Fig. 11, a representative unit cell, which is repeated in all directions, consists of eight woven fibertows in the shape of elliptic cylinders and one parallelepiped that is used to model the interlaminar porosity. Fig. 12 presents the predicted in-plane modulus of the Nicalon/SiC composites versus % porosity. Increased porosity was found to decrease the stiffness, as observed experimentally (Fig. 8). Moreover, there is a reasonably good agreement between the predicted and measured moduli, as could be seen from the Fig. 12. In through-thickness direction, longitudinal moduli predicted by the model considerably varied from the measured longitudinal moduli.

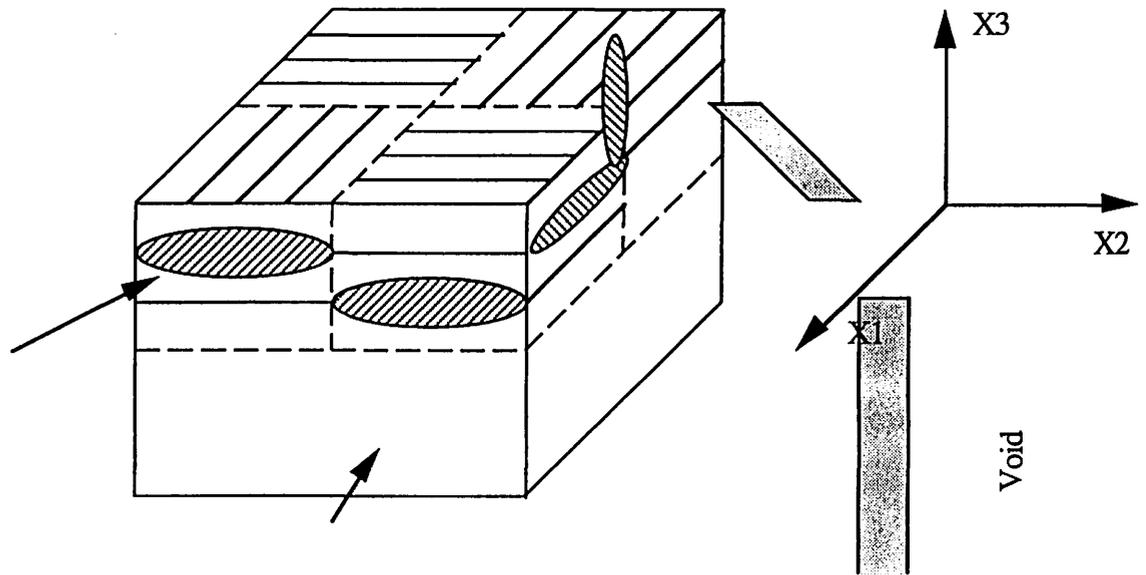


Fig. 11. A model based on periodic structure

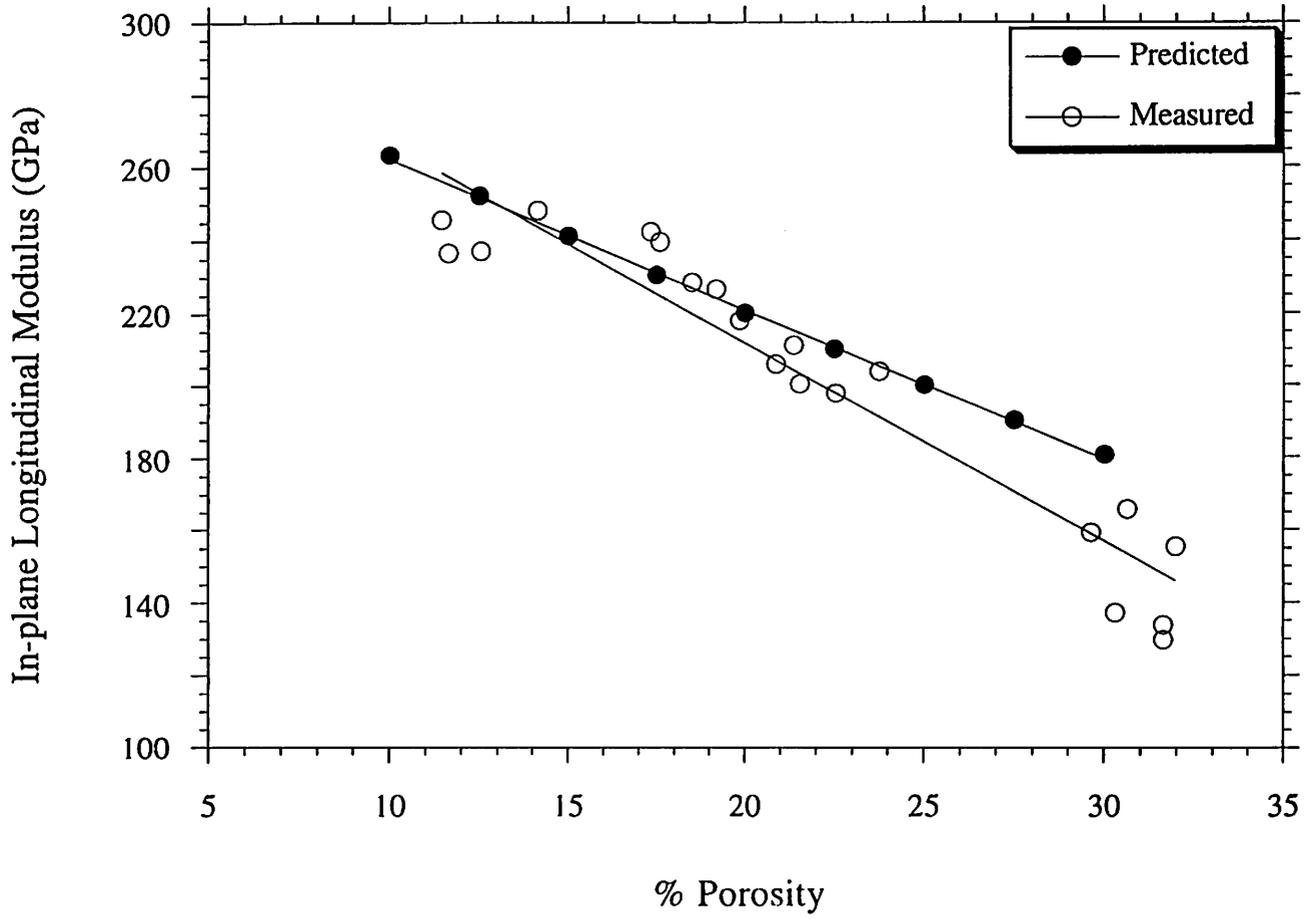


Fig. 12. Comparison of Predicted and Measured In-plane Longitudinal Moduli

Discussion

In this investigation, both through-thickness and in-plane moduli are determined using ultrasonic methods. The through-thickness moduli are found to be much less than the in-plane moduli. This trend could be related to the greater amount of interlaminar porosity than the porosity at the fiber tows intersection, as shown in Fig. 6. Increased porosity significantly decreased both in-plane and through-thickness moduli.

A periodic model using a homogenization method is formulated to predict the influence of porosity on the moduli of woven fabric composites. The theoretical prediction showed the decreased moduli with increased porosity. Moreover, the in-plane moduli are predicted to be in reasonably good agreement with the experimental results. Nevertheless, the predicted moduli are found to be somewhat greater than the experimental results. In the formulation of the theoretical model, only the [0/90] plies are considered without the inclusion of the off-angle plies of [30/120] and [60/150]. The inclusion of the off-angle plies in the theoretical model could alter the differences between the predicted and measured moduli.

Conclusions

Nicalon fiber-reinforced ceramic-matrix composites were fabricated by infiltrating low-density fibrous structures with gases that decompose and deposit as SiC to form the matrix using the Forced Chemical Vapor Infiltration (FCVI) technique at Oak Ridge National Laboratory (ORNL). Both through-thickness and in-plane moduli were measured using ultrasonic methods. The in-plane moduli were found to be greater than the through-thickness moduli. Increased porosity was found to significantly decrease both through-thickness and in-plane moduli. A theoretical model developed to predict the effect of porosity on the moduli of woven fabric composites predicted the decreased moduli with increased porosity. The predicted moduli were found to be in reasonably good agreement with the experimental values.

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