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LASER ULTRASONIC MEASUREMENTS ON CERAMIC MATERIALS WITH ROUGH SURFACES

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At the Idaho National Engineering Laboratory, noncontacting ultrasonic measurements are being made on materials while they are being processed at elevated temperatures. These materials have rough surfaces, typical of those encountered during processing. A pulsed laser is used for generation of elastic waves and a laser Doppler interferometer for detection. Processes being studied are the fabrication of silicon carbide fiber reinforced silicon carbide (SiC-SiC) by chemical vapor infiltration (CVI) and the sintering of zinc oxide (ZnO). For SiC-SiC produced by CVI, the velocity and wave amplitude of elastic waves propagating through the sample have been found to depend on the porosity[1] and on the fiber-matrix interface bond strength. Surface waves on this material are affected by the density of the near surface layer, which can be substantially greater than that of the interior for many CVI processes. Data are also presented for the sintering of ZnO, where the laser ultrasonic method is used to monitor density and shrinkage in real time.

A confocal Fabry-Perot interferometer, described in References 2 and 3, is used for detection. It is sensitive to the Doppler shift of the light reflected from the sample surface, which is moving due to the ultrasonic wave. An argon ion continuous laser (maximum output of about 1 W in the 514 nm green line) illuminates the sample surface. Rough surfaces are particularly challenging for laser detection of ultrasonic waves. However, the Fabry-Perot is sensitive to the frequency of the light collected, not to the phase. Hence, the surface does not have to be optically smooth, and the light can be collected from a relatively large area (about 1 to 4 mm^2) on the surface. This permits higher illumination power to be used, which can be crucial for rough surfaces, without damaging the surface. The elastic waves are generated in the sample by a Q-switched Nd-YAG laser that delivers a 10 ns pulse of up to 300 mJ at 1064 nm. The generation mechanism in the material is thermoelastic conversion of the laser energy to ultrasound. The two lasers can be incident on the same surface or on opposite surfaces, depending upon the application.

The interface bond strength in ceramic composites is important in determining the toughness of the material. For SiC-SiC, the wave shape and velocity of elastic waves propagating through the bulk vary markedly with the degree of bonding between the fibers and the matrix. The through-transmission arrangement used to monitor the bonding is shown in Figure 1. Measurements made on three samples having the same density but deliberately fabricated with different bond strengths are shown in Figure 2. For the weak bond, the elastic wave is dramatically delayed and attenuated, arriving at 5.5 μs, while for the intermediate bond it is only slightly delayed compared to the strong bond case. Work is continuing on developing a quantitative correlation between the bond strength and the shape and velocity of the ultrasonic wave.

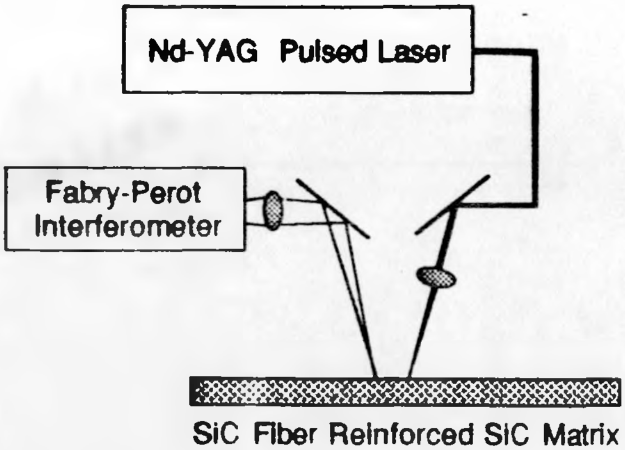

Figure 1. Block diagram for through-transmission elastic waves. The Nd-YAG pulse width is 10 ns at 1064 nm. The interferometer uses an argon ion laser with a maximum of 1 W in the green 514 nm line.

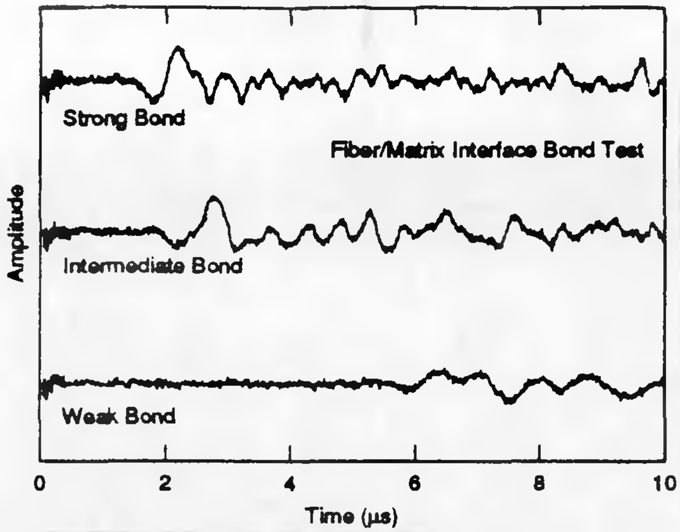

Figure 2. Ultrasonic waveforms for samples of SiC-SiC with different fiber surface treatments prior to infiltration. The ultrasonic wave is markedly delayed and attenuated for the weak bond compared to the strong and intermediate bonds.

CVI processes that do not use pressure and temperature gradients tend to produce materials with higher density near the surfaces than in the interior. Elastic waves traveling between two points on a surface of the composite are dominated by this higher density surface layer, while elastic waves traveling through the composite probe the interior. Combined measurements of these two waves are expected to show the extent of layering in the composite. Thermoelastic expansion, which is responsible for production of the elastic waves, produces surface waves in addition to the waves in the bulk of the material. The arrangement shown in Figure 3 was used to measure surface waves. Waves were generated on a 100 x 38 x 3.3 mm plate of SiC-SiC. These were not strictly surface waves, since the wavelength is comparable to the plate thickness. However, in this layered material, elastic waves propagating across ply are highly attenuated compared with elastic waves propagating along a ply. Hence, the elastic waves at the detector are expected to contain only a small contribution from reflection from the opposite surface of the plate. These elastic waves will, of course, probe the interior as well as the surface of the plate. Figure 4 shows waves generated on the composite and, for comparison, on a 53 x 53 x 26 mm block of monolithic SiC. A 180 mJ, 5 x 10 mm generation pulse was incident 18.5 mm from the detection point, oriented such that the minimum travel distance was 16 mm. The surface wave is seen to arrive later and to be much broader for the composite than for the monolithic sample. The lower wave speed and the loss of the higher frequency components for the composite are expected because the high porosity interior is also being probed. Quantitative correlation of surface properties with surface wave features has not been completed yet, however, these results show that laser ultrasonics can monitor surface conditions through measurement of surface waves. Future use of this technique will allow real-time monitoring of surface properties during CVI.

The properties of sintered ceramics are determined in part by the residual porosity in the material. Monitoring the porosity during the sintering stage could lead to improvements in both the consistency of the properties and the efficiency of the process. Work at the INEL has demonstrated the utility of laser ultrasonics in monitoring sintering. The velocity of elastic waves in ZnO has been shown to be directly related to the sample porosity,[2,3] which provides a direct probe of density during sintering. Figure 5 shows the experimental arrangement used for real-time measurements of the porosity. The ZnO green bodies were 25.4 mm in diameter, 11 mm thick, and 47% of theoretical density. Figure 6 shows the detected ultrasonic waveforms for a green sample, and for the same sample just after sintering has begun and at 5 and 10 minutes into the process. The decrease in transit time for the elastic wave is quite dramatic throughout the sintering process. The recorded ultrasonic waveforms provide the transit time for the elastic wave. If uniform shrinkage is assumed, a good assumption with uniform green density and minimal temperature gradients in the sample, the sample density can be derived from the transit time.[3] The sample density and shrinkage can then be tracked in real-time. Figure 7 shows the results for a sample of ZnO similar to that used for Figure 6. The density and shrinkage were both calculated from the transit time to an accuracy of 2-4%.

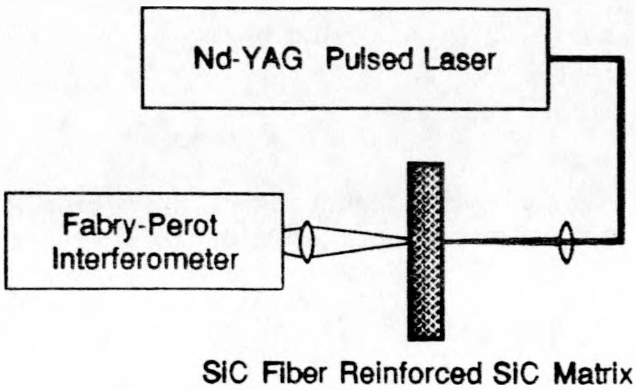

Figure 3. Block diagram for measurements with surface waves is similar to Figure 1 except that generation and detection occur on the same surface.

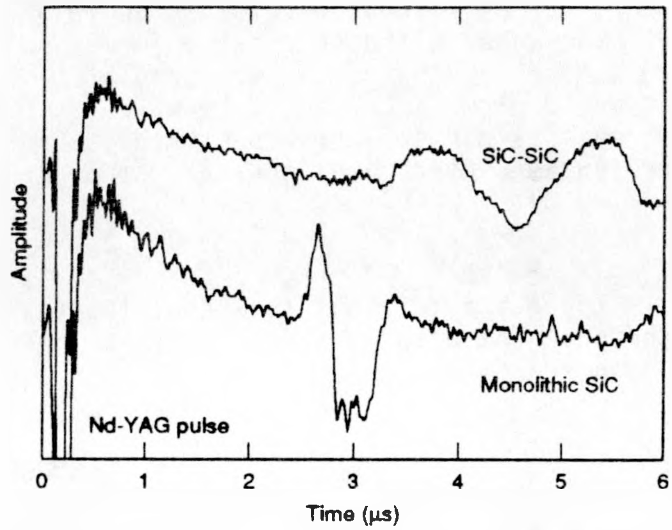

Figure 4. The surface wave on the composite, SiC-SiC, is broader and delayed compared to the surface wave on the monolithic sample.

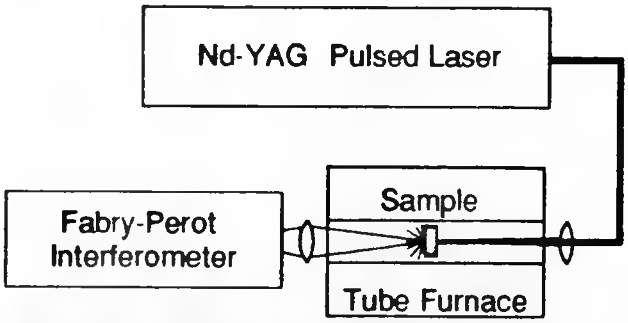

Figure 5. Block diagram for monitoring sintering is similar to Figure 1 except that the sample is located inside a tube furnace.

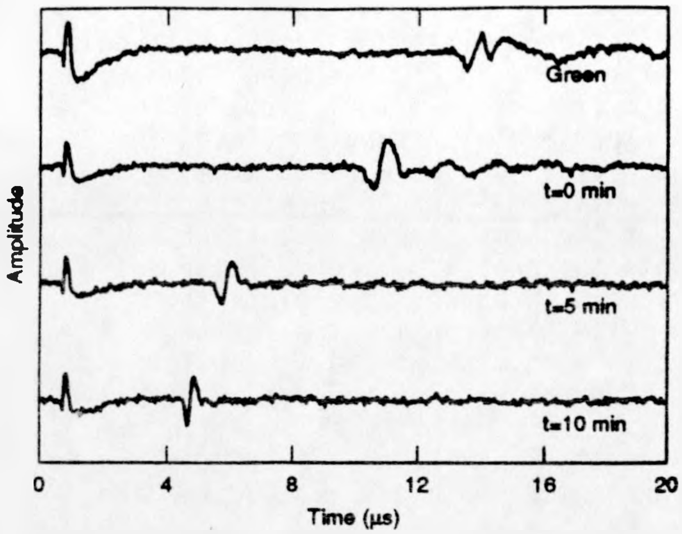

Figure 6. Ultrasonic waveforms recorded with the laser ultrasonic setup for a ZnO compact in the green state and at 3 times during sintering. The wave becomes sharper and arrives markedly earlier as sintering progresses.

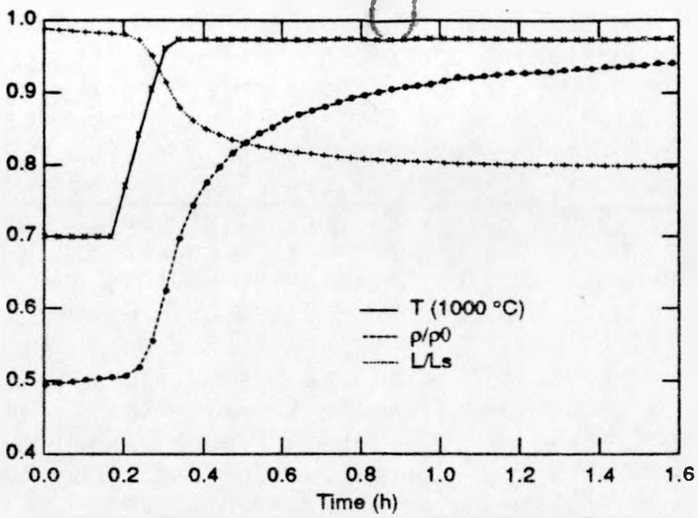

Figure 7. Real-time record of shrinkage (L/Ls) and density (ρ/ρ_0) of a ZnO compact during sintering. Values were computed from the transit time of the ultrasonic pulse assuming uniform shrinkage.

These applications show that laser ultrasonics with the Fabry-Perot detection scheme can be used in several ways to monitor the processing of ceramics *in situ* and in real time.

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