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NONINTRUSIVE ULTRASONIC SENSOR FOR MONITORING AND CONTROL OF ELECTROCONSOLIDATION® PROCESS

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Abstract. Electroconsolidation® is a proprietary process for rapid pressure-assisted densification of complex-shaped parts made from powder preforms. The resistive heating used in this method allows extremely high temperatures to be generated rapidly. This paper describes an ultrasonic pitch-catch technique for nonintrusive measurement of temperature within the die cell; this technique can be used for precise control of densification and sintering of parts. Results of ultrasonic data for heating trials conducted at up to 2000°C indicate that average axial temperature in the die can be predicted within 3%.

INTRODUCTION

The Electroconsolidation® (EC) process is a new proprietary method for rapid pressure-assisted densification of preformed materials into complex-shaped parts by simultaneous application of pressure and heat [1]. The key advantages of this method compared to established methods such as hot isostatic pressing include (a) the ability to densify complex-shaped parts to near net shape without the need to clad the part, (b) capability to apply high temperatures up to 3000°C, (c) rapid cycle times, (d) flexibility to introduce multiple parts, and (e) most important, low cost. It can be used to manufacture parts from powders of many different materials, including metals, ceramics, and polymeric composite materials.

Figure 1 is a schematic diagram of the basic apparatus used in the EC process. The part to be densified is immersed within a bed of free-flowing, electrically conducting granular medium within a cylindrical die chamber. Pressure is applied uniaxially by double-acting hydraulic rams from the top and bottom of the die chamber. Heat is generated resistively within the granular medium by passing electrical current through it from a low-voltage, high-current DC power supply by a water-cooled copper electrode on each of the two low-resistance rams. The ideal pressure-transmitting medium should be free-flowing and electrically conducting, yet more resistive than the rest of the circuit to act as resistive heat source; it should also be chemically inert and stable at high temperature. Graphitic carbon powder (75- 500 μm) is one such material that closely meets these above requirements and is used as the pressure-transmitting medium in our work.

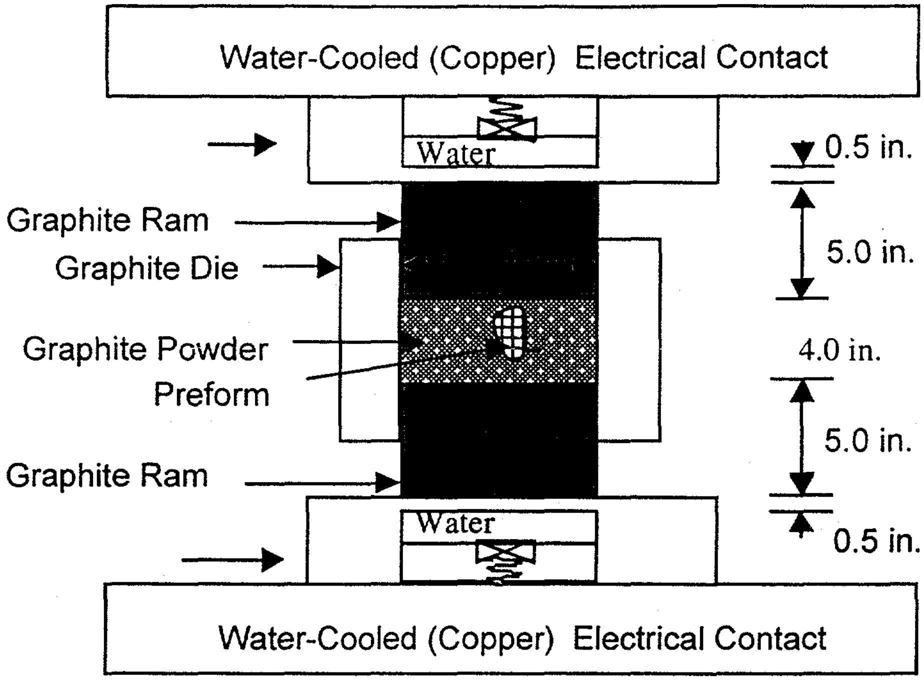

FIGURE 1. Schematic diagram of Electroconsolidation® apparatus; position of pitch-catch transducer assembly, along with typical dimensions of the die, is also shown.

Because the electrical properties of the graphite powder medium vary with pressure and temperature, wide temperature fluctuations can occur within the die chamber. Moreover, the geometry and location of the preforms, as well as their electrical properties, will affect the electric field distribution and hence the temperature profiles within the die. A real-time nonintrusive temperature sensor is needed for proper control of the temperature during sintering and to avoid over- or under-heating of the preform. Thermocouples are not practical in an EC production environment because of their intrusive nature and are not suitable above 2000°C. This paper describes a nonintrusive, real-time ultrasonic pitch-catch sensor that is capable of predicting average temperature along the axial direction in the EC die at up to 3000°C.

ULTRASONIC PITCH-CATCH SENSOR

The velocity of sound whether in a solid, liquid, or gas, varies with temperature in a predictable manner [2,3]. It increases with temperature in gases, but decreases with temperature in most solids and liquids. Based on this property, high-temperature thermometric measurements have been made in hostile environments such as combustion furnaces [4]. The ultrasonic time-domain reflectometer, for example, uses a thin rod with one or more notches along its length, and the temperature between the notches or between a notch and the end of the rod can be inferred by measuring with a pulse-echo method, the round-trip travel time of sound in the notched segment [5]. Although high temperatures can be measured this way using appropriate sensor material, the approach still suffers because it is intrusive, like thermocouples.

In the ultrasonic pitch-catch technique, the transmitting and receiving transducers are located outside the process die and thus do not interfere with the process. The ideal location for the transducers in the EC apparatus is between the water-cooled electrical contact and the graphite ram at the top and bottom of the die, as shown in Fig. 1. Measuring the transit time of pulsed ultrasound along the line of sight between the transducers and knowing the path lengths, we can calculate the velocity of sound in the powder bed of the die. Because sound velocity in a particulate medium is a function of both pressure and temperature, our approach is to measure the velocity of sound in the graphite bed as a function of temperature at different pressures and heating rates, and to then correlate the ultrasound data with temperature under certain operating conditions.

Factors that govern the operation of the ultrasonic sensor include transducer temperature, coupling between interfaces, and wave propagation characteristics in the particulate medium. The temperature at the transducer face must stay below its design limit of 130°C, even though the internal temperature in the process can reach up to 3000°C. To protect the transducer from the process heat, the transducer is placed over a circulating water bath within a water-cooled copper block. As shown in Fig. 1, the ultrasonic waves must propagate through different sections of materials and encounter several interfaces, starting from the transmitter transducer, continuing through copper, water, copper, solid graphite, graphite powder, solid graphite, copper, water, copper, and ending with the receiver transducer. Although an oil-based couplant is used between the transducer and the water chamber where the temperature is low, the coupling mechanism between the parallel faces of the transducer assembly and the graphite ram, where temperature can be high, is simply pressure. Wave scattering and attenuation occur in the particulate medium; the scattering depends on the wavelength relative to particle size and the attenuation increases with the void spaces between the particles in the graphite bed. Transmitting ultrasonic energy through the graphite bed with adequate signal-to-noise ratio would therefore require the use of either low frequency and/or application of moderate pressure to the bed.

Figure 2 shows schematically the instrumentation used for the ultrasonic pitch-catch data collection. A 500 kHz tone burst signal from a function generator (Wavetek) is amplified by a 40 dB RF power amplifier (EIN) and applied to a 500kHz, 1in.-diameter transmitting transducer (Panametrics). The signal from an identical receiving transducer is amplified and filtered by a 40 dB preamplifier with a band-pass filter set at 450-550 kHz. The received signal is displayed on a digital oscilloscope (LeCroy) with respect to the trigger signal of the transmitter waveform. Total transit time between the pitch and catch signal is recorded corresponding to the location of the first peak of the received waveform.

TESTING OF SENSOR IN THE ELECTROCONSOLIDATION SYSTEM

A pilot EC facility is in operation at Argonne National Laboratory as part of a National Institute of Standards and Technology's Advanced Technology Program to develop and demonstrate the commercial application of the process. It comprises a 200-ton hydraulic press, a 10,000 ampere DC power source, and various die and ram sets for working with preforms with a maximum dimension of 4 inch.

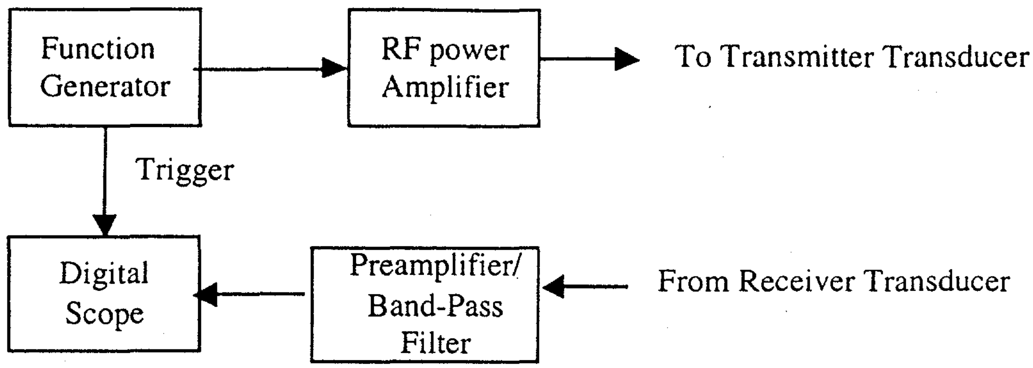

FIGURE 2. Schematic diagram of ultrasonic pitch-catch sensor electronics.

An EC experiment starts with loading the die chamber manually with graphite powder and the preform between the upper and lower rams; this assembly is then placed between the water-cooled copper blocks. The hydraulic rods apply the initial pressure. At ambient pressure, the receiver transducer does not pick up any transmitted signal because of excessive attenuation in the particulate bed. As the pressure is increased, we begin to receive the transmitted ultrasonic pulse, typically above 500 psi; and at ≈1000 psi, the signal-to-noise level becomes adequate for temperature tests. Pressures of 2000–4000 psi are generally used and are usually applied at the onset of heating. The pressure can be held constant or ramped up or down during the heating/cooling cycle. Power is applied and the current is adjusted from 1500 to 10,000 A to establish a prescribed heating rate of up to 2000°C per minute.

Typical operational variables of the EC system are die diameter, ram pressure, heating rate, and hold time. The ultrasonic sensor was tested against the various test conditions listed in Table 1. To calibrate the sensor, Type C thermocouples were placed at different locations: center of the bed, within the ram, outside surface of the die, and on the transducer surface.

RESULTS AND DISCUSSION

The main variable that was monitored in all the ultrasonic tests was the total time of flight (TOF) of ultrasonic pulses along the line of sight between the two transducer locations shown in Fig. 1, although the amplitude of the received pulses was also checked for excessive attenuation of sound in the materials during heating. Tracing the path of ultrasonic waves, it becomes obvious that total TOF is mainly constituted by the transit times in the solid graphite rams (typically 5 in. long; two sections) and in the graphite powder bed (typically 4 in. thick). We separately measured the ultrasonic TOF of the top and bottom rams at ambient temperature by using a pulse-echo technique. These times are then subtracted from the total TOF to obtain the TOF in the bed. Knowing the bed height, which is obtained from a pair of position transducers instrumented on the rams, we can calculate the velocity of sound in the graphite bed by dividing the bed height by the TOF in the bed.

Because the TOF in the rams also changes with temperature in a heating experiment, the calculated velocity of sound based on the ambient-temperature TOF

Table 1. Test matrix for the ultrasonic tests in the Electroconsolidation system.

Test No.	Die Diameter (in.)	Pressure (psi)	Preform Included?	Heating Rate (°C/min)	Maximum Temperature (°C)
1	5	500 to 5000	No	N/A	Ambient
2	3	2500	No	50	1630
3	3	2500	No	50	800
4	3	2500	No	50	800
5	3	2500	No	50	800
6	5	1500	No	50	1650
7	5	2000	No	100	1800
8	5	2000	Yes	50	2000
9	5	2000 & 4000	Yes	100	2000

data for the rams is only approximate. To analyze the magnitude of this error, we designed and performed a heating experiment of up to 700°C with a thin (0.5-in.) bed in which the bed effect was small. In this case, the total TOF still decreased with temperature indicating that the velocity of sound in the ram increased with temperature; but the change in TOF with temperature was only a small fraction (0.15) of the typical changes observed in a 4-in. bed. Also, because the ram is in contact with the water-cooled copper on the one end and the hot bed on the other, the average temperature rise in the ram is much lower than in the bed. As a result, we neglected the temperature effect in the ram in the ensuing analyses.

We first conducted a pressure test (Test 1) to determine its effect on the velocity of sound in the bed. Because of the powder compaction, the velocity of sound increased with pressure as shown in Fig. 3; signal height also increased with pressure due to reduced attenuation. This clearly indicates that we must keep the pressure constant to determine the effect of temperature on the velocity of sound.

In the next four experiments (Tests 2-5) with a 3in. die, we held the pressure constant at 2000 psi and monitored the TOF as we increased heat at a fixed rate of 50°C/min. With an aim to determine the ultrasonic properties of the bed in these initial experiments, we did not use preforms because they could modify the velocity of sound. Also, we used a fresh batch of graphite powder in each of these experiments to eliminate any hysteresis effect. The total TOF showed a steady decrease during heating, and the velocity of sound calculation for the bed showed similar slopes for all cases. However, the starting points, namely the velocity values at ambient temperature, were different among the tests; this may be due to how the particles were initially packed in the bed. Using as the reference the value corresponding to that of 50°C in Test 2, we corrected the offsets in the velocity curves of Tests 3-5. Figure 4 gives the velocity of sound versus temperature curves for the four tests. Remarkably,

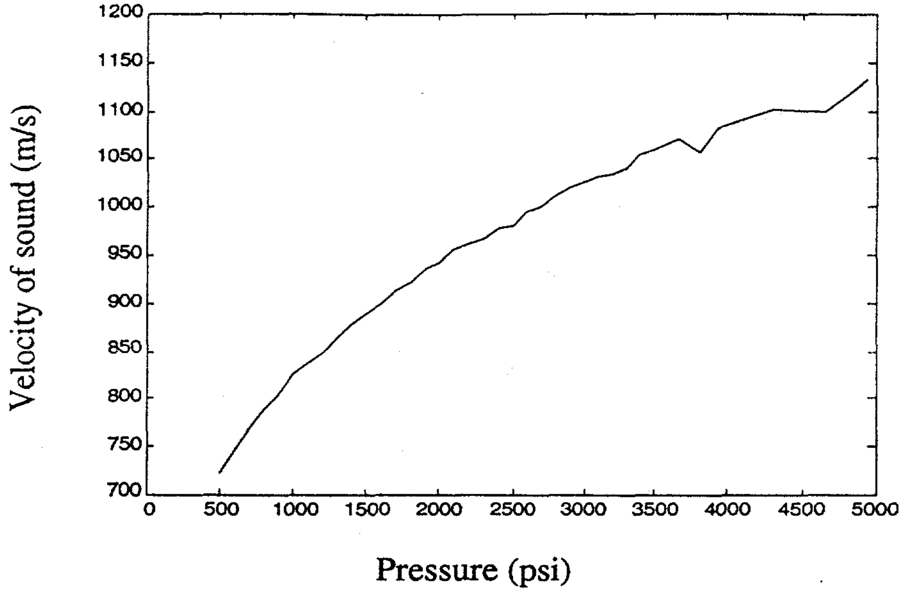

FIGURE 3. Change of velocity of sound in graphite bed with pressure at ambient temperature.

the data for the four separate experiments are reproducible; they follow a quadratic curve fit as shown by the solid line in Fig. 4. An error analysis with respect to the curve fit indicates that the temperature can be predicted within an uncertainty of 3%.

In the next four experiments (Tests 6-9), we used a 5-in. die, different pressures, and two heating rates, and we tested with and without preforms. For the

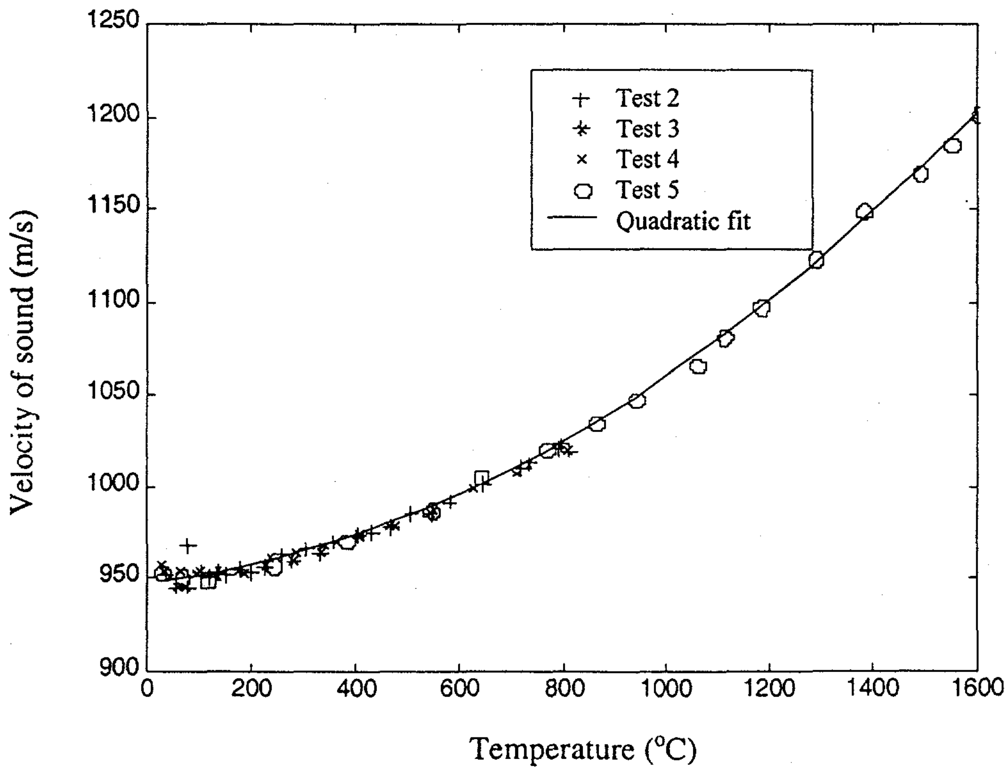

FIGURE 4. Calibration curve for a 3-inch die at 50°C/min heating rate.

preform, we used a 90% dense SiC disk, 1 in. diameter and 0.19 in. thick. In Test 9, we even used a pressure step of 2000 psi up to 850°C and 4000 psi thereafter up to 2000°C. Because a step change in pressure introduces a step change in the velocity of sound, we corrected the offset between different pressures with reference to a value corresponding to 2000 psi. Figure 5 shows the offset-corrected plots of heating and cooling curves for Tests 6-9. Note that the heating curves are grouped according to the heating rate and that the slopes of the 50°C/min heating curves are higher than those of the 100°C/min curves. This change in slope with the heating rate may be due to the error involved in our calculation of the bed TOF.

Consistently, there was a strong hysteresis between the heating and cooling phase in the first heating-cooling cycle, but as seen in Test 6 corresponding to 1500 psi, this nearly disappeared in the second heating-cooling cycle. The hysteresis is attributed mostly to changes in the elastic and relaxation properties of the powder with temperature cycling and also partly to the different rates of heating and cooling. However, from the process control standpoint, the reproducibility of velocity data during the first heating phase will suffice. Figure 6 gives a quadratic calibration curve derived from Tests 6 and 7 for the 5in. die at a 50°C/min heating rate. Error analysis reveals that temperature can be predicted with this method with an uncertainty of <2.2 percent for temperatures up to 2000°C in a 5in. die.

The heating experiments were terminated at ≈2000°C, mainly because of thermocouple limitations; during this time, the surface temperature at the transducer

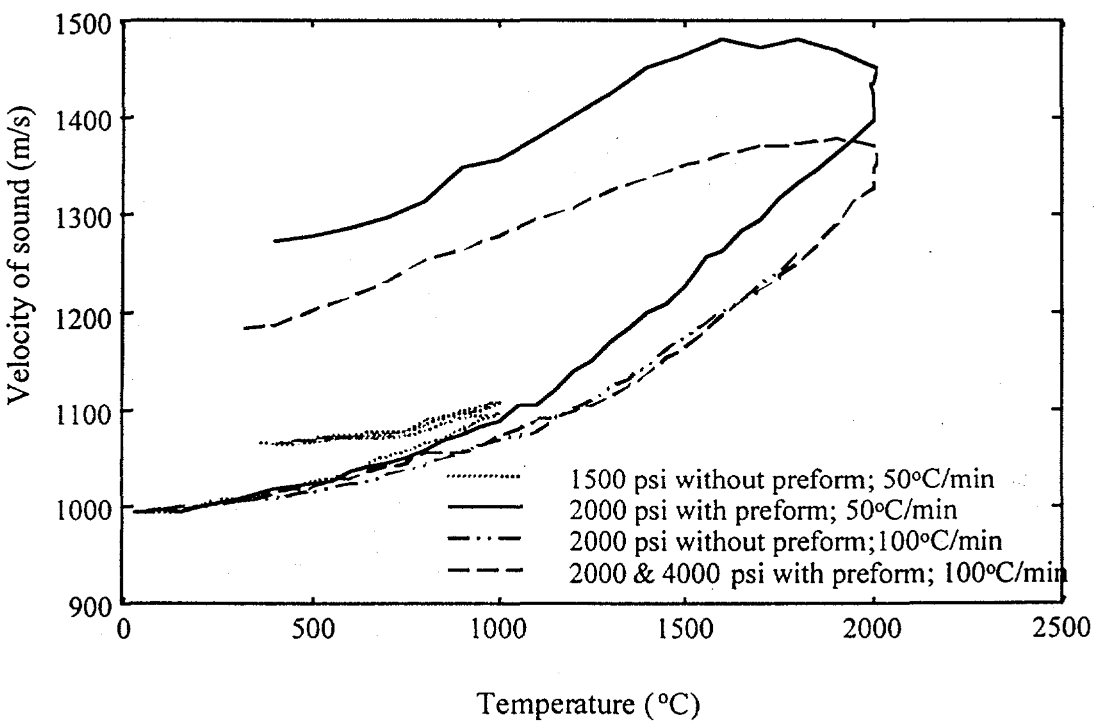

FIGURE 5. Change of ultrasonic velocity with temperature in graphite bed at fixed pressures and heating rates.

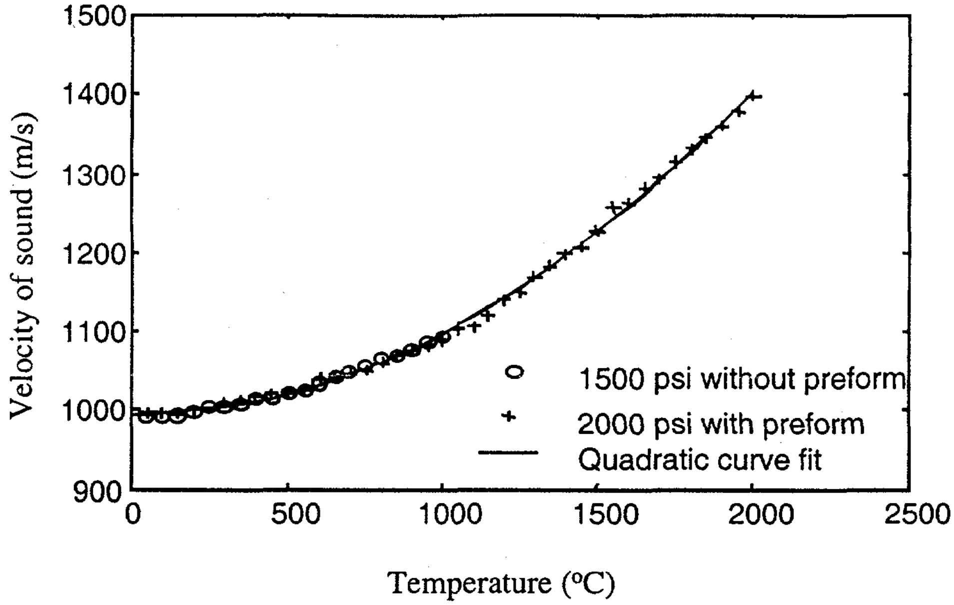

FIGURE 6. Calibration curve for a 5-inch die at 50°C/min heating rate.

reached only 64°C. Considering that the transducers can withstand up to 130°C, we believe that the sensor can be used to monitor and control the temperature in the Electroconsolidation system up to the target value of 3000°C.

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