Pacific Northwest NATIONAL LABORATORY

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Sound Speed as a Candidate for Internal Temperature Monitoring During Solid Phase Processing of Materials

October 2020

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U.S. DEPARTMENT OF ENERGY

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Abstract

Internal temperature is an important parameter for understanding the solid phase processing (SPP) of materials and improving feedback control of these processes. Friction stir processes (FSP), shear assisted processing and extrusion (ShAPE™), and ultrahigh velocity (UHV) cold spray are examples of such processes. Simple thermal models based on temperatures measured at the boundary of the deforming body cannot capture the dynamic nature of temperature generation within the deforming solid. One possibly way of understanding internal temperature is by measuring sound speed, which varies as a function of temperature for materials and directing ultrasonic wave propagation through selected material volumes of interest. In this effort, longitudinal wave speed is estimated for aluminum 6061 in a configuration amendable to ShAPE. Piezoelectric materials bonded to a 25.4-mm thick specimen provided data from which longitudinal wave speed was estimated. The linear relationship of wave speed versus temperature is discussed and how an inverse function might be used is postulated; that is, wave speed measurements could be used to estimate the internal temperature of selected material volumes for feedback during SPP of materials. Specimen configuration, instrumentation, and data and analysis are reviewed.

Summary

Longitudinal wave speed was estimated from ultrasonic measurements acquired from a 2.5 cm thick, aluminum (AL) 6061 specimen over the temperature range of 23 °C – 400 °C. A decreasing linear relation was observed in the data with a slope of -0.0011 mm/(μs °C) and linear regression R^2 value of 0.992. The good fit to a linear function inferred an inverse relation might be used to estimate temperature by a measurement of longitudinal wave speed. Ultrasonic data was acquired with lead metaniobate elements, attached to the AL specimen. Each of the two elements had a 3.1 mm diameter and a 15 MHz resonance frequency. Pulse-echo and through-transmission techniques were used to acquire data. The piezoelectric sensors became dysfunctional above 400 °C as they approached the Curie temperature of 460 °C for lead metaniobate (K-81) (Piezo Technologies 2018). One data acquisition pass of increasing temperature was acquired. Data essentially duplicated the reported linear slopes of -0.0091 mm/(μs °C) and -0.00093 mm/(μs °C) for AL 6061 over the temperature ranges, respectively of 0 °C – 200 °C (Asay and Guenther 1967, 4087) and 0 °C – 100 °C (Christman, et al 1971, 21). The specimen configuration was selected to replicate a typical displacement between the ShAPE container and mandrel (Whalen et al. 2017, 316) and interrogation of a material region adjacent to the extrusion die where heat is generated by SPP. Future work should examine the following:

- Higher temperature piezoelectric material such as a modified bismuth titanate which has a Curie temperature of 770 °C (Meggitt plc n.d.)
- Other wave modes such as shear waves and surface waves to examine acoustic parameters that may have potential for feedback control
- Techniques to interrogate selected material regions of interest
- Repeat measurements since only one data acquisition pass with increasing temperature was acquired.

ShAPE implementation will naturally require ultrasonic techniques that couple ultrasound with high-temperature, rotating billet material. One candidate is electromagnetic acoustic transducers (EMATs) that can be configured for high temperature. Advantages of EMATs are the passage of ultrasound directly into and out of the high-temperature billet material (Boyd and Sperline 1988, A-7, B-10, and B-17) and elimination of additional couplant material typically used by piezoelectric techniques (Innerspec Technologies, Inc. n.d.).

Acknowledgments

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Acronyms and Abbreviations

AL	Aluminum
CL or C_L	Longitudinal Wave Speed
DOE	Department of Energy
EMAT	Electromagnetic Acoustic Transducer
FSP	Friction Stir Processing
LBU	Laser Based Ultrasound
L-Wave	Longitudinal Wave
MS	Microsoft Corporation
PNL	Pacific Northwest Laboratory
PNNL	Pacific Northwest National Laboratory
ShAPE	Shear Assisted Processing and Extrusion
SPP	Solid Phase Processing
SPPSi	Solid Phase Processing Science Initiative
UHV	Ultrahigh Velocity

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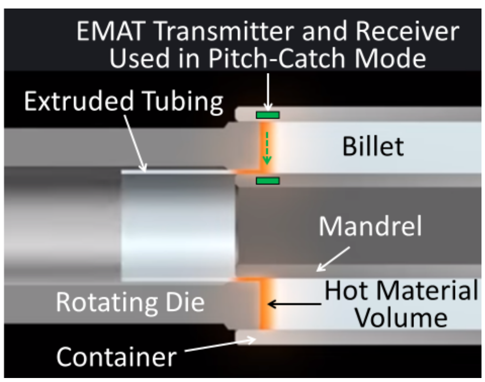
1.0 Introduction

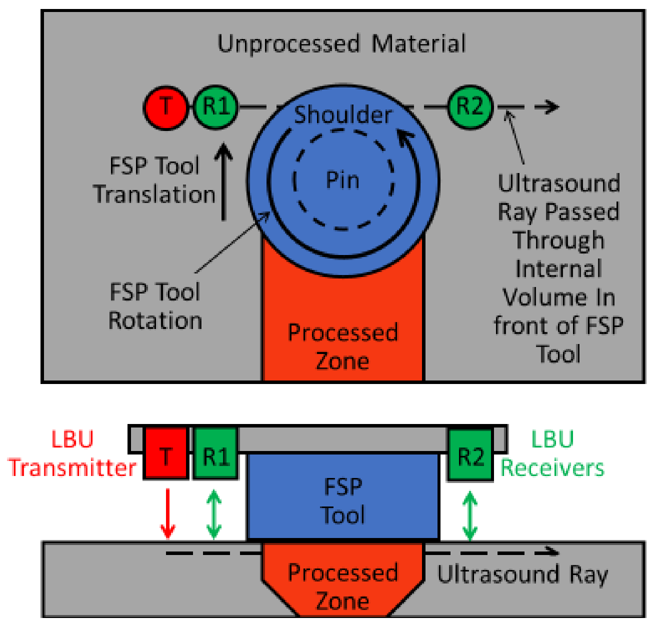
Internal temperature is an important parameter for understanding the solid phase processing (SPP) of materials and improving feedback control of these processes[1]. Friction stir processes (FSP), shear assisted processing and extrusion (ShAPE™), and ultrahigh velocity (UHV) cold spray are examples of such processes. Simple thermal models based on temperatures measured at the boundary of the deforming body cannot capture the dynamic nature of temperature generation within the deforming solid. One possible way of understanding internal temperature is by measuring sound speed and attenuation which vary as a function of temperature for materials and directing ultrasonic wave propagation through selected material volumes of interest (Lynnworth 1989, 41, 240-241, 370, 394-398).

Concepts of utilizing ultrasonic interrogation of selected material volumes for ShAPE and FSP are shown in Figure 1. Note that sensitivity to a selected region of the working material volume is accomplished by placement of ultrasonic transmitters and receivers. Transduction techniques such a piezoelectric material, electromagnetic acoustic transducers (EMATs), and laser based ultrasound (LBU) can be customized to be amenable to a SPP process.

EMATs may be advantageous for ShAPE since access is difficult and direct line of sight does not exist due to material opaqueness. A thought is that ceramic sight windows could be placed in the container and mandrel with the two EMAT sensors facing each other as shown in Figure 1.A. The interrogated material volume is that of the material between the two EMATs.



A. ShAPE: The SPP working material volume is contained between the container and mandrel. EMATs can access the SPP working material volume by electromagnetic induction and use of ceramic windows.



B. FSP: The SPP working material volume surrounds the rotating pin. LBU can access the SPP with surface waves generated by laser ultrasound since line of sight exists around the tool. A standoff sensing technique such as LBU may be advantageous around rotating tools.

Figure 1. Future vision of temperature sensors configured for ShAPE and FSP.

[1]Cynthia Powel. 2019. FY2020 Call for Seedling Concept Papers, MS PowerPoint presentation. Richland, WA: Pacific Northwest National Laboratory.

LBU may be advantageous for FSP since the material surface is visually accessible and laser beams could be directed to the locations shown in Figure 1.B. The generated ultrasonic surface wave from the transmitter would then pass across the surface and be detected by two receivers. LBU also has the advantage that standoff generation and reception of ultrasonic waves simplifies usage around a spinning FSP pin. Depth of penetration of a surface wave is approximately one wavelength; e.g., approximately 3 mm for a 1 MHz Rayleigh wave.

Sound speed and attenuation are dependent upon multiple parameters such as the following:

- Material properties, material phase, texture (Goebbels 1980, 87-115)
- Stress state by means of the acoustoelastic effect (Hughes and Kelly, 1953, 1145-1149) and (Castellano et al. 2017, 1524-1525)
- Material temperature (Lynnworth 1989, 41, 240-241, 370, 394-398).

Examples of direct measurement of wave speed as a function of temperature for multiple steel alloys was accomplished with EMATs (Boyd and Sperline 1988, A-7, B-10, and B-17).

2.0 Specimen Configuration

An aluminum (AL) specimen was prepared in a manner characteristic of some ShAPE applications to examine sound speed as a candidate for internal temperature monitoring during SPP. As shown if Figure 1.A., a billet wall thickness of approximately 2.5 cm was selected. Piezoelectric elements were used instead of EMATs due to their common usage, flexibility of use, and the availability of piezoelectric materials and supporting ultrasonic instrumentation. An AL alloy was selected for the specimen due to the lower processing temperature of AL relative to other alloy families such as steel and the ease of experimental planning for lower temperature measurements.

3.0 Instrumentation

Two longitudinal wave (L-wave) piezoelectric elements were bonded to an AL 6061 specimen with Cotronics Resbond™ 904 zirconia ceramic adhesive and electrodes with Cotronics Resbond 931C graphite adhesive, Figure 2. A modified lead metaniobate (K-81) was the piezoelectric material with a 3.1 mm diameter and 15 MHz nominal frequency. One element was an ultrasonic transmitter in pulse-echo mode and the other element mounted on the opposite side was a receiver in through-transmission mode. High temperature, mineral insulated coaxial cables were used between piezoelectric elements and ultrasonic instrumentation. A thin gold wire electrically bridged the positive electrode surface of the piezoelectric element and the center conductor of the high temperature coaxial cable. Two Omega TJ36-CAIN-116U-12-CC K-type thermocouples were inserted into close fitting holes to a mid-thickness depth of the AL specimen to monitor interior temperature of the specimen.

The AL specimen was placed in a laboratory oven and the thermocouples and high temperature coaxial cables were feed through a small portal opening at the top of the oven. Argon gas was feed into the oven chamber to minimize oxidation of the graphite adhesive.

Label Key

- A Fume Hood
- B Laboratory Oven with Top Portal Opening
- C Thermocouple Reader
- D Thermocouple Monitor
- E Ultrasonic Transmitter and Pulse-Echo Receiver (JSR Ultrasonics DPR300)
- F Ultrasonic Through-Transmission Receiver
- G Digital Oscilloscope (Ultrasonic Data)
- H AL-6061 Specimen in Laboratory Oven
- I Piezoelectric Element, Lead Metaniobate (K-81)
- J Mineral Insolation, Coaxial Cables J1 and J2
- K Gold Wire (Bridge between Cable Center Conductor and Piezoelectric +Electrode)
- L Thermocouples L1 and L2
- J Argon supply tube

Figure 2. Instrumentation and enlargements of inner chamber of laboratory oven and AL specimen with mounted piezoelectric elements and thermocouples.

4.0 Data and Analysis

Thermocouple data and ultrasonic responses of the pulse-echo and through-transmission techniques were recorded at discrete temperature settings. Relative arrival time shifts for the pulse-echo and through-transmission waves were tracked using a zero-crossing technique for the respective responses. Arrival time was estimated by subtracting the time duration between the zero-crossing fiducial feature and the earlier 10% amplitude signal envelop threshold obtained from room temperature data. Data were acquired with one temperature data acquisition run from room temperature to 400 °C with the oven set to discrete values at temperature intervals of 50 °C. Temperature stabilized but still increased slightly during acquisition of ultrasonic data. Three measurements were made at each target temperature value. The piezoelectric sensors became dysfunctional above 400 °C as they approached the Curie temperature of 460 °C for K-81 (Piezo Technologies 2018). Specimen thickness was measured with calipers multiple times with an average 25.45 mm thickness and changes due to thermal expansion were estimated with a linear thermal expansion coefficient of 2.32 x 10^{-5}/°C.

Plots of sound speed versus temperature were acquired for the pulse-echo response and the through-transmission response with MS Excel, Figure 3.

Decreasing linear relations were observed in the data with estimated slopes of -0.0011 mm/(μs °C) and -0.0010 mm/(μs °C) for pulse-echo and through-transmission, respectively. This essentially duplicated reported slope values of -0.00091 mm/(μs °C) (Asay and Guenther 1967, 4087) and -0.00093 mm/(μs °C) (Christman, et al 1971, 21).

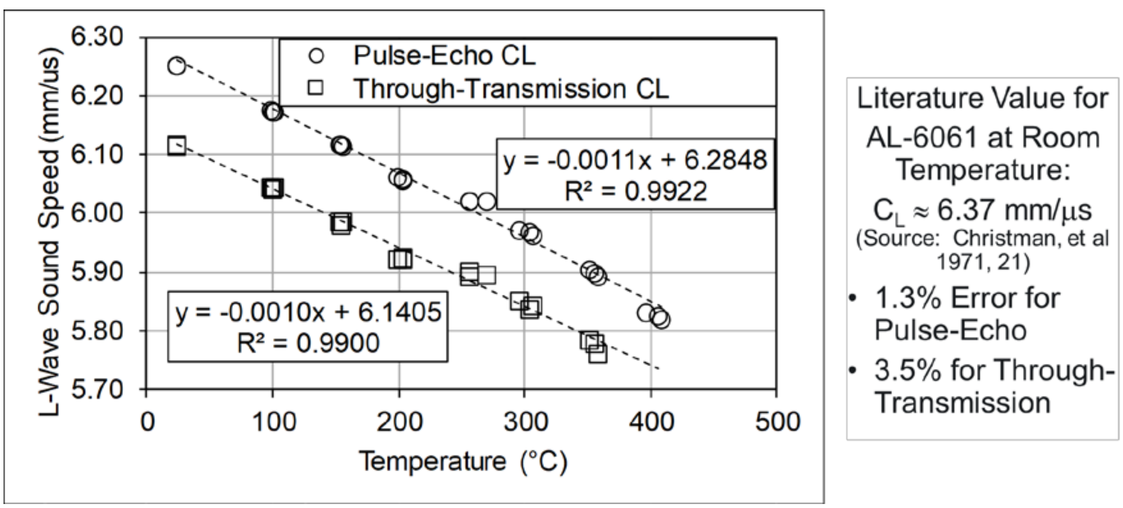


Figure 3. Longitudinal wave speed as a function of temperature was estimated with one data acquisition run with temperature increasing from 23 °C to 400 °C.

5.0 Discussion

An AL specimen configuration was selected to replicate a typical displacement between the container and mandrel of a ShAPE unit (Whalen et al. 2017, 316). A displacement of 2.5 cm was believed to be representative. One concept was that an ultrasonic transmitter and a receiver could be placed opposite each other with one near the exterior surface of the billet in the ShAPE container wall and the other near the interior surface of the billet in the ShAPE mandrel wall. Both could be placed near the rotating die where material work is performed, and high temperatures are created. Since the piezoelectric elements had a 3.1 mm diameter, the interrogated material volume was essentially an internal cylinder with a 3.1 mm diameter and a 2.54 cm length. The estimated wave speed is an integration process over the entire cylindrical volume. A decreasing linear function was observed in the data with an estimated slope of -0.0011 mm/(μs °C) and a small standard deviation indicated by a trendline R^2 value of 0.992. The change of L-wave sound speed over the 400 °C interval is relatively large compared to value variation away from the trendline. This indicates sensitivity and that the inverse process of measuring L-wave speed and estimating billet material temperature is feasible.

Recommendations for future work are as follows:

- Examine higher temperature piezoelectric material such as a modified bismuth titanate which has a upper working temperature of 650 °C and a Curie temperature of 770 °C (Meggitt plc n.d., 1-2)
- Repeat measurements since only one data acquisition run was acquired and refine the technique to increase measurement accuracy
- Examine other wave modes such as shear waves and surface waves to examine acoustic parameters that may have potential for feedback control
- Examine techniques to interrogate selected material regions of interest.

ShAPE implementation will naturally require ultrasonic techniques that couple ultrasound to high temperature, rotating billet material. One candidate technology is EMATs (Innerspec Technologies, Inc. n.d.). EMATs can be configured for high temperature and efficiently pass ultrasound into and out of billet material without the need of added coupling material which is typical of piezoelectric techniques (Boyd and Sperline 1988, A-7, B-10, and B-17).

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