IN-SITU CHARACTERIZATION OF CAST STAINLESS STEEL MICROSTRUCTURES

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ABSTRACT
Cast austenitic stainless steel (CASS) was commonly used in selected designs of nuclear power reactor systems for corrosion resistance and enhanced durability in service. CASS materials are generally coarse-grained and elastically anisotropic in nature, and are consequently difficult to inspect ultrasonically, largely due to detrimental effects of ultrasonic wave interactions with the coarse-grain microstructures. To address the inspection needs for these materials, new approaches that are robust to these phenomena are being developed. However, to enhance the probability of detecting flaws, knowledge of the microstructure and the corresponding acoustic properties of the material may be required. This paper discusses the application of ultrasonic backscatter measurement methods for classifying the microstructure of CASS components, when making measurements from the outside surface of the pipe or component. Results to date from laboratory experiments demonstrate the potential of these measurements to classify the material type of CASS for two homogeneous microstructures—equiaxed-grain material or columnar-grain material. Measurements on mixed or banded microstructures also show correlation with the estimated volume-fraction of columnar grains in the material. However, several operational issues will need to be addressed prior to applying this method for in-situ characterization of CASS microstructure.

INTRODUCTION
Cast austenitic stainless steel (CASS) that was commonly used in selected designs of U.S. nuclear power plants is a coarse-grained elastically anisotropic material. The engineering properties of CASS made it a material of choice for these nuclear power reactor systems. However, the fabrication processes resulted in a variety of microstructures that are difficult to inspect ultrasonically [1-4], largely due to detrimental effects of wave interactions with the coarse-grain microstructure inherent to this class of materials [4-7]. The interaction of ultrasonic waves with CASS material results in phenomena such as sound-speed variations, ultrasonic beam re-direction and partitioning, high attenuation and high background acoustic noise caused by scattering, and phase variations across a wave front. These phenomena make reliable and effective ultrasonic inspections of CASS materials highly challenging.

To address CASS inspection needs, new approaches are being developed that are more robust to these phenomena and potentially result in improved probability of detection and characterization of flaws. However, these methods may require knowledge of the microstructure and the corresponding acoustic properties of the material [4, 7, 8]. Characterization of the microstructure of the component can potentially improve the general inspectability of a component by optimizing ultrasonic inspection parameters for CASS components, such as optimized ultrasonic phased-array inspection systems [9, 10], and in assisting interpretation of measured ultrasonic data. The characterization of CASS microstructure must be done in-situ, to enable dynamic selection and optimization of the ultrasonic inspection technique.

Previous efforts at Pacific Northwest National Laboratory (PNNL) on in-situ ultrasonic techniques for microstructure characterization examined longitudinal wave attenuation, normal incidence longitudinal wave backscattering, and ultrasonic diffuse fields [11-13], with a focus on pure microstructures (equiaxed and columnar grains). This paper presents the results of scoping experiments that investigate the potential application of longitudinal wave backscattering methods as a function of incidence angle for in-situ classification and/or characterization of material microstructures in CASS piping, when making

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1 This work was sponsored by the U.S. Nuclear Regulatory Commission under U.S. Department of Energy Contract DE-AC05-76RL01830; NRC JCN N6398; Mr. Wallace E. Norris, Program Monitor.
measurements from the outside surface of the pipe. The focus of the present study was to evaluate ultrasonic backscattering on a wider range of microstructures and determine if responses from known microstructures can be differentiated.

ULTRASONIC SCATTERING
Generally, in coarse-grained materials, acoustic wave propagation is a function of the microstructure, frequency, and wave mode [4, 7, 14-17]. Depending on the frequency (or, equivalently, wavelength) and mode of the acoustic wave, a range of behaviors may be observed. Theoretical studies based on the use of both semi-analytical models and numerical models have been performed for better understanding the behavior of ultrasonic wave propagation in coarse-grained materials [15, 18-23]. Documented phenomena include beam deviation or skew [4, 7, 14, 16, 23], phase distortion of the acoustic wave front [15, 24, 25], and attenuation [12, 14].

As acoustic waves interact with materials, scattering of energy occurs at interfaces such as grain boundaries. Scattering in the direction of the transmitting transducer is often referred to as backscatter. However, the scattered energy at any angle (relative to the transmit direction) can be measured, if a receiver can be placed appropriately. Scattering typically occurs if the mean scatterer (grain) size ($D$) is comparable to the wavelength $\lambda$ of the acoustic wave, and a change in acoustic impedance is present across the grain boundary [26]. The contrast in acoustic impedance across a grain boundary can occur due to anisotropy of the elastic properties of grains and the different orientation of each grain [15]. In general, the scattering behavior of ultrasonic waves from single scatterers may be broadly classed into three regimes [17]—Rayleigh, stochastic, and geometric—depending on the mean grain size ($D$) relative to the wavelength of the acoustic wave used. In CASS materials, the grain sizes can vary over a large range [27] and, therefore, the scattering behavior may range from Rayleigh to geometric regimes in a single material volume. Ultrasonic backscatter measurements have been used for a range of material characterization applications [17]. The backscattered signal can be correlated with grain size [28, 29], and in characterization of texture and orientation in transversely isotropic welds [30].

The ability to time-gate a signal and select backscatter from a specific region in the material may make backscattering attractive for CASS microstructure characterization as a function of position in the material volume. However, in materials with strong multiple scattering, the backscattered measurement may only be indicative of bulk properties. Further, in coarse-grained materials, the ultrasonic beam may interact with only a few grains prior to reflection from the back surface [31], again resulting in measurements that are indicative of bulk properties. Finally, in CASS materials (or in anisotropic inhomogeneous materials in general), the beam redirection and partitioning that is likely to occur, particularly with angle-beam incidence, may make microstructure characterization using angle-beam incidence difficult, if the goal is to classify microstructure as a function of depth/location.

Given these constraints, the choice of wave mode and frequency (or, equivalently, wavelength) for scattering measurements becomes critical. Assuming that an appropriate frequency (or frequencies) can be identified, the hypothesis is that polycrystalline equiaxed microstructures should generally be correlated with higher backscattered energy (notwithstanding the angle of incidence relative to the surface of the specimen) due to the coarse-grained nature of the microstructure, the random orientation of grains, and the typically rather large ratio of grain size-to-wavelength ($D/\lambda$). Higher frequencies ($D/\lambda \gg 1$, stochastic and geometric scattering [17]) will likely result in multiple scattering and a correspondingly higher backscatter signal. For columnar grains with incidence along the grain axis (which corresponds to incidence normal to the outer surface), the combination of a lower attenuation and preferential wave propagation direction should, theoretically, result in a lower overall backscatter measurement. However, with incidence at an angle relative to the columnar grain growth direction, the backscattering measurement will likely resemble that from equiaxed grains.
METHODS

Measurement Approach

A series of ultrasonic backscattering experiments were performed using CASS specimens. Table 1 summarizes the different specimens used in this study, along with the microstructure (grain structures and sizes) in each of these specimens.

The experimental setup used a RITEC (RITEC, Inc., Warwick, Rhode Island) square-wave pulser (model SP-801), broadband receiver (model BR-640), diplexer (RDX-2), and a focused immersion ultrasonic transducer. Three separate transducers were used to collect backscatter measurements. The first operated at 2.25 MHz (Panametrics V395, 38.1-mm diameter, 230-mm focal length). The second transducer was a 1-MHz transducer (Panametrics A392S, 38.1-mm diameter, 152.4-mm focal length), while the third was a 5-MHz transducer (Panametrics A307R, 25.4-mm diameter, 230-mm focal length). The transducers were operated in pulse-echo mode. The angle of incidence (at the center of the transducer) varied from 0 degrees to 25 degrees to the outer surface of the specimen. A simulation using Imagine3D\(^2\) (Version 2.5) was conducted to identify the defocus distance that would ensure that the ultrasonic energy was focused approximately on the back-surface. This information was used to determine placement of the transducer above the outer surface of the specimen. The transducer was positioned to ensure that the same volume of material was insonified, regardless of the angle of incidence. A mechanical scanner was used to record the backscatter signal over a 12.7-mm × 12.7-mm region, with a step size of 0.254 mm. Data was acquired using a 16-bit computer-controlled analog-to-digital converter (Gage Compuscope CS82g), using a sampling rate of 50 MHz. At every position, 16 waveforms were averaged to improve the signal-to-noise ratio. The recorded A-scan contained the backscatter information from an acoustic path length corresponding to a full-V inspection path. The instrument settings (receiver gain and receiver bandwidth) were adjusted to enable optimal backscatter measurement, depending on the specimen and microstructure being investigated. However, the analysis normalized the recorded data to a constant gain and bandwidth setting (gain of 56 dB and bandwidth depending on transducer center frequency). This enabled a direct comparison of the measurements at a single frequency from the different specimens.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Layer Thicknesses (mm)</th>
<th>Microstructure in Each Layer</th>
<th>Grain MLE (mm)</th>
<th>Min Grain Size (mm)</th>
<th>Max Grain Size (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>APE1</td>
<td>14, 29, 27</td>
<td>Eqa, Eqa, Eqa</td>
<td>1.5, 1.8, 2</td>
<td>0.44</td>
<td>8.86</td>
</tr>
<tr>
<td>MPE-6</td>
<td>24, 25, 22</td>
<td>Col, Eqa, Eqa</td>
<td>2.3, 2.2, 2.3</td>
<td>0.56</td>
<td>26.8</td>
</tr>
<tr>
<td>OPE-2</td>
<td>30, 21</td>
<td>Col, Eqa</td>
<td>1.1</td>
<td>0.21</td>
<td>16.7</td>
</tr>
<tr>
<td>B511-C</td>
<td>60</td>
<td>Col</td>
<td>2.5</td>
<td>0.6</td>
<td>12</td>
</tr>
<tr>
<td>B511-E</td>
<td>58</td>
<td>Eqa</td>
<td>2.3</td>
<td>0.6</td>
<td>7</td>
</tr>
<tr>
<td>AAD-2</td>
<td>34, 13, 18</td>
<td>Col, Mix, Eqa</td>
<td>1.5, 2.4</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Note: Eqa = equiaxed; Col = columnar, Mix = mix of columnar and equiaxed

The analysis algorithm used the following procedure.

\(^2\) http://www.utex.com
1. For each position of the transducer, the backscattered energy was computed by first squaring the A-scan data, and then low-pass filtering the result. Squaring the signal shifts the frequency content of the measurement towards the baseband, and also replicates it at a higher frequency (twice the center frequency of the transducer). A low-pass filter is therefore an effective means of extracting the energy (the envelope) of the signal. While smaller cutoff frequencies (on the order of several kHz) can be used to extract the envelope, the use of spatial averaging in this instance also achieves a similar goal. Therefore, for the data in this study, a cut-off frequency equal to the transducer center frequency was used.

2. The computed backscatter energy signals from a region around the transducer position were averaged. While the size of this neighborhood region can vary depending on the application, in this study, the neighborhood size was selected to be 12.7 mm × 12.7 mm, corresponding to the entire scan size.

3. The resulting backscattered energy response between the front surface and back-surface reflections was used as the input to a least-squares curve fitting algorithm. The assumed form of the backscatter energy was

\[ y = Ae^{-\alpha x}, \]  

where \( y \) is the backscattered energy, \( x \) is the distance (acoustic path length), \( A \) is the (unknown) intensity proportional to the scattering coefficient, and \( \alpha \) is the (unknown) decay coefficient for the backscattering energy. This expression assumes a single scattering scenario \[31\] in an isotropic homogeneous medium.

The measurements in this study were taken with the transducer focused approximately on the back surface and no diffraction correction was applied. The need for diffraction correction of the backscattering measurements from this experimental setup needs to be studied further.

RESULTS
Figure 1 shows an example of backscattered energy at 2.25 MHz using the averaging procedure for specimen APE-1. The data are presented using a logarithmic scale to enhance differences. The data in these figures show the variation in backscattered energy as a function of incidence angle. As seen from these figures, as the angle of incidence is increased, the amount of backscattered energy increases. Moreover, the arrival time of the peak scattered energy shifts \[32\] to the right, corresponding to a delay. Additionally, as the angle of incidence changes from the normal, the reflected signal from the back wall is no longer in the direction of the transducer, and therefore, the back-wall signal diminishes as the angle of incidence increases. The curve, described by Eq. (1), was fitted to the backscattered energy, and the parameters \( A \) and \( \alpha \) extracted from the curve-fitting procedure. The parameter \( \alpha \), which is equivalent to the rate of decay of the backscatter energy as a function of acoustic path length, is plotted as a function of frequency in Figure 2. The data presented in Figure 2 corresponds to normal incidence backscatter only. The variation of this parameter as a function of angle of incidence is plotted in Figure 3.

Analysis of the data also indicated that in all CASS specimens tested, the backscattered energy deviates from a pure exponential decay (after about 2–3 cm into the material). This behavior is indicative of multiple scattering \[15, 31\].

As seen from Figures 2 and 3, the computed backscatter decay coefficient \( \alpha \) for normal incidence longitudinal waves showed a correlation with microstructure. In particular, equiaxed-grain material exhibited higher backscatter than columnar-grain material at 2.25 MHz (corresponding to a lower decay coefficient) (Figure 3). The backscattered energy also appears to be a function of the average grain size. The backscatter measurements also indicated a strong propensity towards multiple scattering \[26, 31\] at 2.25 MHz and 5 MHz.

As the angle of incidence increases (from zero degrees or normal incidence), the random orientation of the grains in equiaxed microstructures is likely to result in no significant change in backscattered energy, because the number of interfaces (boundaries) between grains that interact with the applied energy will not change on average with incidence angle. In purely columnar microstructures, or in microstructures with an outer
Figure 1 – Backscattered energy as a function of incidence angle for one specimen (logarithmic plot). The horizontal axis shows acoustic path length in meters (assuming a nominal wave speed of 5800 m/s in CASS).

Figure 2 – Parameter for normal incidence, as a function of frequency, obtained from exponential curve fitting to backscattered energy.
columnar microstructure band, normal incidence on the outside surface of the pipe or component corresponds to incidence along the grain growth direction (or the main axis of the grain), resulting in smaller backscattering energy due to the small number of grain boundaries interacting with the applied energy. With increasing angle of incidence, an increased number of grain interfaces interact with the applied ultrasonic energy, increasing the backscatter. Offsetting this increase is the higher amount of mode-converted energy (from longitudinal to shear wave), potential beam splitting and skewing, and a reduction in backscatter. The net result is a backscattered energy profile that is very similar to those from equiaxed microstructures as the angle of incidence increases past 5 or 10 degrees (Figure 3). The apparent increase in $\alpha$ at 2.25 MHz (decrease in backscattered energy) as the incidence angle increases from 0 to 5 degrees could be due to several factors, including a grain growth direction that is not normal to the surface, and experimental error in aligning the transducer with the (curved) outside surface of the specimen. In specimens with mixed or banded microstructures (OPE-2 and MPE-06) where one or more of the bands have columnar microstructure, the backscattering behavior is seen to be between those from pure columnar and pure equiaxed microstructures.

Analysis as a function of frequency (Figure 2) shows that backscatter measurements at 1 MHz did not show any significant differences between the two microstructure categories. This is likely due to the grain sizes in the CCSS specimens chosen for this study. The mean grain sizes (as measured on a single plane that was polished and etched) ranged from about 1.2 mm to about 4 mm [33]. Assuming that these mean grain sizes are representative and using a wavelength of 5.8 mm (at 1 MHz, in stainless steel), the ratio of mean grain size-to-wavelength $D/\lambda$ is less than 1. Thus, it is likely that the material appears to be somewhat homogeneous at this frequency, with smaller backscatter, and little difference in the scattering behavior between the different microstructures. Further, the specimen thickness was close to an integral multiple of the wavelength at 1 MHz, and therefore this frequency may be close to a resonant frequency of the specimens examined.

As the frequency increases (wavelength decreases), the ratio of grain size relative to wavelength increases. At 2.25 MHz, $D/\lambda$ is between about 0.5 and 2, and strong multiple scattering is a possibility. At 5 MHz, $D/\lambda > 2$ resulting in increased backscattering (decrease in $\alpha$) and little difference in the backscattering behavior between the different microstructural classes. In particular, the increased scattering is seen to be relatively independent of incidence angle and not clearly correlated to microstructure class.

CONCLUSIONS
This paper summarizes proof-of-concept studies of ultrasonic backscattering measurements for classification and/or characterization of material microstructures in CASS piping materials from the outside surface. The results demonstrated the potential of ultrasonic backscattering measurements (as a function of incidence angle) to potentially classify diverse CASS microstructures (including pure equiaxed
or columnar, and mixed/banded microstructures). The experiments provided promising results and demonstrated a reasonable basis to believe that, with further refinement of these techniques, real-time in-situ classification of CASS materials is feasible.

An advantage for ultrasonic backscatter measurements is the potential for microstructure classification as a function of thickness. However, the studies conducted to date did not analyze the backscatter coefficients as a function of location in the specimen. Instead, the measurements were used to provide an average (over the specimen thickness) estimate of backscattered energy in each specimen. An examination of the backscattering measurements indicated that anomalies or changes in microstructure (i.e., banding) through the specimen thickness may be detectable when using normal incidence longitudinal backscatter measurements. However, further development of this technique (including measurements from different angles of incidence) may be necessary to fully characterize the acoustic properties (such as acoustic impedance and grain size) of the material [34].

A potential issue with the use of ultrasonic techniques for microstructure characterization is the selection of an appropriate frequency. Ideally, frequency selection would ensure that the ultrasonic wave has significant measurable interactions with the microstructure. In most cases, this requires the selection of higher frequencies (shorter wavelengths) so that significant multiple scattering occurs. In this respect, the requirements are opposite those of inspection for flaw detection, where the goal is minimal interaction of the ultrasonic wave with the microstructure. In practice, because in-situ grain sizes will be unknown, it is likely that multiple frequencies will be needed for field characterization of CASS materials.

There are several factors that will need to be addressed before this technique can be applied in a field setting. Some of the variables used in the calculations (such as the phase velocity used to estimate the acoustic path length in backscattering) may not be accurately known for in-situ field settings. Some experimental variables such as ultrasonic coupling must also be controlled carefully to obtain reliable measurements. Finally, under field conditions it may be more useful to compute backscatter parameters relative to known microstructures to enable in-situ classification of grain structure.

Further development of this technique for in-situ measurements on CASS will need to focus on refining the measurement protocols and analysis tools, including evaluating algorithms for assessing CASS microstructural parameters (such as mean grain size and acoustic impedance variations) and verifying results using additional specimens that ideally are representative of the majority of microstructures that may be encountered in legacy U.S. nuclear power plant CASS components.

REFERENCES


