ABSTRACT

JOHNSON, JOSEPH ALLEN. High-Temperature Yttrium Calcium Oxyborate Acoustic Emission Sensor. (Under the direction of Dr. Xiaoning Jiang).

This thesis details the design, prototyping, and testing of a high temperature yttrium calcium oxyborate acoustic emission sensor. This sensor took advantage of a shear mode yttrium calcium oxyborate piezoelectric crystal to detect Lamb waves in a thin bar substrate. These Lamb waves were induced via two methods: Hsu-Nielsen test and fatigue cracking. The Hsu-Nielsen test, which consists of breaking a pencil lead on the substrate, created a cheap and reproducible acoustic emission source with which to test the detection capabilities of the sensor. The fatigue crack test was used as a more realistic method of replicating stress waves that would be detected in industry. The signal produced by the sensor for each of these tests was analyzed in both the time and frequency domains. The time domain signals were inspected to show the high and low frequency Lamb wave components, and these signals were transformed into the frequency domain using Fast Fourier Transform techniques. The frequency components of the Lamb waves induced in each test displayed additional information about the capability of the sensor. The frequency and time relationship was analyzed through a spectrogram, which could be related directly to the dispersion curves of the lamb waves. The ability of the sensor to detect both symmetric and antisymmetric mode Lamb waves displayed the capabilities of the sensor up to 1000 °C.
High-Temperature Yttrium Calcium Oxyborate Acoustic Emission Sensor

by
Joseph Allen Johnson

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2013

APPROVED BY:

_______________________________  ________________________________
Xiaoning Jiang                    Fuh-Gwo Yuan
Committee Chair

______________________________
Yun Jing
DEDICATION

To my greatest support, my wife Krystal.
BIOGRAPHY

Joseph Johnson was born and raised in Lansing, NC and graduated from Ashe County High School in 2007. He received his B.S. in Mechanical Engineering from North Carolina State University in spring 2011. In fall 2012 Joseph began pursuing his master’s degree under the direction of Dr. Xiaoning Jiang after spending a year working in industry.
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I. INTRODUCTION

A. Acoustic Emission Background

Acoustic emission (AE) testing is a rapidly growing method for testing a broad spectrum of wear and fatigue in mechanical, aerospace and civil structures. This sensing method originated from the field of structure monitoring in civil engineering and has the unique ability to evaluate an entire structure and locate a discontinuity as it forms and propagates. The earliest recognition of acoustic emission arose in the field of metallurgy, with the working of tin and steel. In the 1930’s acoustic emission was tested for and recorded in Japan with wood under stress; in Germany with the martensitic transformation of steel; and in the US with micro-seismic activity in mines. [1] The next major leaps in acoustic emission testing came with compressive load tests in concrete. Ruesch related acoustic emission to the volumetric change of concrete in the 1960’s, and in conjunction with several other papers on the subject and the development on standard procedures and devices for testing, the first acoustic emission groups were formed in the 1970’s. [1]

Acoustic emission is usually caused by a stress-induced deformation and involves the release of energy from a localized source within a material, which takes the form of an elastic wave [2]. A visualization of this event is shown in Figure 1.
The deformation that is most associated with acoustic emission in research and industry is fatigue cracking, although the waves can be induced by friction wear, corrosion and other forms of deformation [3]. The diverse AE source mechanisms each create signals that are dependent on the source, as well as the geometry of the substrate. As more studies are completed on the characterization of acoustic emission signals in academia and industry, frequency and amplitude characteristics can be found for varying sources. The amplitude and frequency characteristics for different types of wear have been found, and can be used to determine the severity of wear. Adhesive wear, or wear at a nanometer surface finish level, tends to emit higher frequency and higher amplitude signals than abrasive wear, which is at a micrometer level and is accompanied with shavings. [4] Wear frequencies and amplitudes can also be compared with crack acoustic emission characteristics, which is shown in Figure 2 in a comparison graph from Hase et al.
Since cracking is used in the experiments for this thesis, broadband signals of frequency less than 500 kHz should be expected. Understanding of the acoustic wave properties that are emitted from a particular source is vital for proper equipment selection. Insensitivity to low amplitude or high frequency would obviously inhibit a sensor’s capabilities. Likewise, environment weighs heavily on selection, especially since many
industrial or commercial machines are associated with hot and dirty surroundings. Considering these factors, many different sensor mechanisms have been investigated, including surface capacitive sensors, laser sensors, and piezoelectric sensors. Capacitive sensors have an incredible sensitivity range, but limited robustness due to a high sensitivity to electromagnetic noise [5]. Laser sensors, on the other hand, have an inherent robustness because they do not have to be in contact with the subject structure, but this leads to a lack in sensitivity that is necessary for early detection of stress waves [6].

Robustness and sensitivity are both critical properties of an acoustic emission sensor, so a method that can provide both is necessary. Piezoelectric crystal, which generates an electric charge when subjected to a mechanical stress, is the most common AE sensor [6]. These sensors can have a very high level of sensitivity, and the wide variety of crystal material properties demonstrates that piezoelectrics can be easily adapted for many different applications, providing the necessary robustness.

B. High temperature acoustic emission sensing needs

In order to enhance the sensitivity, acoustic emission transducers need to be as close to the high stress region of a machine as possible due to the attenuation of elastic waves in material [7]. However, the high-stress areas in a nuclear reactor, turbine, or internal combustion engine reach temperatures well above critical temperatures for conventional piezoelectric materials [8, 9]. Acoustic emission sensors have been used in industry with a waveguide, or a rod or bar that is fastened to the structure and the sensor to keep the crystal
relatively cool. This introduces complexities when attempted to read the waveform and frequency response of an acoustic wave for characteristics. [10] Therefore novel sensors developed for high temperature applications need to be capable of direct contact.

Direct contact AE sensors offer great value in nuclear facility structural health monitoring. Nuclear facilities operate on a 40-year license, and since the year 2000, 44 commercial plants have received 20 year extensions on their license (as of 2007) [11]. As the average age of operating nuclear reactors continues to grow, issues such as fatigue stress become more prominent. Fatigue cracking in concrete support structures can be difficult to detect, especially during operating conditions, which can result in compromised structural integrity and halting of operations [12]. Therefore a form of continuous structural health monitoring near the hot regions of a reactor, where fatigue is more likely to occur, is crucial for safe and continuous operation. The operating ranges of nuclear reactors are dependent on their design, but advanced high-temperature reactors use coolant with exit temperatures in the 700 – 1000 °C range, and high temperature gas cooled reactors are designed to supply heat at temperatures up to 950 °C [13; 14]. The effect of these temperature ranges, in addition to harsh environment, has been studied on structural components of reactors, showing changes in the chemical make-up and the expected oxidation [14,15]. These corrosive reactions emit acoustic emission, but the change in chemical make-up can produce unexpected deformities as well. Radioactive materials likewise produce acoustic emission signals, as demonstrated by Stoev et al [16].
Combustion engines likewise provide an application for high temperature AE sensing. Recent research has shown the possibility of using acoustic emission for sensing fuel injector faults in diesel engines, although further research is needed for use in commercial or industrial applications. [17] Acoustic emission has also been studied for internal combustion engines in conjunction with other common health monitoring techniques, such as pressure and vibration sensing, showing the value introduced by the early and sensitive detection of stress waves. [18] The temperature ranges of internal combustion engines do not reach that of nuclear reactors, but may reach temperatures above 650 °C, making current high temperature acoustic emission sensors available in industry unfeasible. [19]

Currently available sensors and materials are not operable in these applications. Limitations include loss of piezoelectric properties and limited electrical properties at high temperatures, chemical degradation or corrosion, and limited availability of materials in bulk quantities. In this thesis, high temperature materials are presented, and a sensor design using yttrium calcium oxyborate (YCOB) is exhibited. The sensor developed for this thesis exhibits sensitivity to multiple Lamb wave modes, with little degradation to the quality of the signal as a function of temperature. The analysis techniques for verifying the ability of the AE sensor are outlined, and details are given for modal analysis techniques of Lamb waves. This method is used to qualify the capabilities of the YCOB sensor at both room temperature and elevated temperatures. The ability to detect low order wave modes is compared to that of Lead Pb(Mg,Nb)O3-PbTiO3 (PMN-PT), which exhibits much higher magnitude piezoelectric properties.
In Chapter 2 high temperature piezoelectric materials are discussed, and further details are given on the material chosen, YCOB. The analysis techniques used to verify the feasibility of the sensor are discussed in Chapter 3, and the sensor design and experimental methods are explained in Chapter 4, respectively. The results of these experiments are exhibited in Chapter 5 using the analysis techniques shown in Chapter 3, as well as the discussion of the results, implications, and areas for future research. Chapter 6 concludes the thesis with an overview of the results.
II. HIGH TEMPERATURE PIEZOELECTRIC MATERIALS

Many factors contribute to the selection of a material for a high temperature sensor. The material obviously must be chemically stable across the desired temperature range, without any phase transition, melting point, or critically altering temperature below the application temperature. For the goals of this thesis, the material must be capable of withstanding exposure to temperatures up to and above 1000 °C without suffering a critical loss in sensitivity. It must also have thermally stable electromechanical properties, such as low dielectric losses and low losses to mechanical quality. All of these factors contribute to the selection of a stable material at high temperatures. [10]

Piezoelectric materials are generally limited in their applications by their phase transition temperature or melting point. Quartz and gallium phosphate piezocrystals suffer from a phase change well below 1000 °C, while tourmaline crystals melt at temperature in the range of 1000-1200°C, leading to the loss of piezoelectricity [20]. Lithium Niobate has been tested in high temperature environments with promising results, but suffers from oxygen losses and resulting decrement in in quality factor [10,21]. Aluminum Nitride film has also performed well as a sensing material at high temperatures, but it is severely limited due to the difficulty of mass production [22]. Langasite crystals have been extensively studied for high temperature applications, but are limited by low quality factor and resistivity at high temperatures. [23] Rare earth calcium oxyboarte single crystals ReCa₄O(BO₃)₃ (ReCOB, Re: rare earth elements such as Gd, La, and Y) have recently been investigated for
use in high temperature sensors. Yttrium calcium oxyborate (YCa$_4$O(BO$_3$)$_3$, YCOB) is one of the promising ReCOB materials due to its high resistivity at elevated temperatures, and its relatively stable electromechanical and piezoelectric properties across a broad temperature range [20]. These materials are shown in Table 1 along with their tested temperature range as an acoustic emission sensor, and their limiting characteristics. [20-24].

Table 1: High Temperature Piezoelectric Sensor Materials

<table>
<thead>
<tr>
<th>Piezoelectric Material</th>
<th>Temperature Range (°C)</th>
<th>Advantages</th>
<th>Limiting Factor (at high temperatures)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gallium Phosphate – GaPO$_4$</td>
<td>930</td>
<td>High Mechanical Quality Factor (Q$_m$)</td>
<td>Increased disorder – lowers Q$_m$</td>
</tr>
<tr>
<td>Lithium Niobate – LiNbO$_3$</td>
<td>800</td>
<td>High piezoelectric properties</td>
<td>Chemical Decomposition</td>
</tr>
<tr>
<td>Aluminum Nitride Film - AlN</td>
<td>1150</td>
<td>Ease of incorporation</td>
<td>High quality bulk fabrication</td>
</tr>
<tr>
<td>Langasite - LGS</td>
<td>800</td>
<td>Lack of phase transition</td>
<td>Oxygen diffusion – low resistivity and Q$_m$</td>
</tr>
<tr>
<td>Gadolinium Calcium Oxyborate - GdCOB</td>
<td>&lt;1300</td>
<td>High temperature stability</td>
<td>Decreased electromechanical coupling</td>
</tr>
<tr>
<td>Yttrium Calcium Oxyborate – YCOB</td>
<td>&gt;1300</td>
<td>High temperature stability</td>
<td>Melting point (~1500 °C)</td>
</tr>
</tbody>
</table>

Recently, vibration sensing was demonstrated with YCOB crystals at temperatures up to 1000 °C, due to the absence of phase transition prior to the melting point, being around 1500 °C [25]. YCOB was tested in comparison with LiNbO$_3$ and AlN for high temperature ultrasonic capabilities, which showed little piezoelectric degradation when exposed to long term high temperatures [26]. This is a promising sign for the capabilities of YCOB as an
acoustic emission sensor material. High temperature AE sensors have been developed in industry as well. Two high temperature commercial AE sensors are shown in Table 2, along with a more common AE sensor for temperature range comparison [27, 28].

Table 2: Commercial High Temperature Piezoelectric Sensors

<table>
<thead>
<tr>
<th>Sensor Model</th>
<th>Sensor Type</th>
<th>Frequency/ Frequency Range</th>
<th>Max Operating Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GE B4 GVN</td>
<td>Ultrasonic</td>
<td>4 MHz</td>
<td>250</td>
</tr>
<tr>
<td>PAC S9215</td>
<td>Acoustic Emission</td>
<td>50-650 kHz</td>
<td>540</td>
</tr>
<tr>
<td>PAC R15a</td>
<td>Vibration/AE</td>
<td>50-400 kHz</td>
<td>175</td>
</tr>
</tbody>
</table>

All of the sensors in Table 2 are in a group of similar sensors, which offer alternative characteristics for different applications. The GE sensors use a delay block to help isolate the piezoelectric layer from hot surfaces, which could be an inhibitor if it were used as an AE sensor, since it would alter the waveform detected by the sensor. The sensors offered by PAC are enclosed in an Inconel case, which allows it to be used in nuclear environments by reducing the exposure to radiation. The acoustic emission sensors that are available for applications in high temperature environments are limited by their piezoelectric element and sensor structure. YCOB may supply the sensitivity and stability necessary for the higher temperatures that occur in nuclear and combustion environments.
III. ACOUSTIC EMISSION ANALYSIS

A. Parameter Analysis

Two primary methods for analysis of acoustic emission waves are used in industry, including parameter and waveform analysis. Parameter analysis is the classic form, because of its lack of dependence on computing power and data storage. Parameter analysis takes advantage of wave characteristics related to the source. The components of the wave signal that are analyzed are [29,30]:

- Hit – The number of total waveforms that are detected, or number of envelopes
- Emission count/Ring-down count – The number of peaks present within a Hit or envelope
- Rise-time – The time interval between the first amplitude in the envelope and the peak amplitude
- Amplitude – The peak voltage from the sensor
- Duration – The time interval between the first and last wave peak in the signal
- Energy – Generally defined as the area under the signal envelope

The parametric analysis of a wave is often skewed by noise, and is dependent on the proper calibration of the sensor, but can be completed very quickly and without the need for advanced computing. An example of the parameters drawn from an AE signal is illustrated in Figure 3.
Figure 3: Acoustic Emission Signal Parameters. [29]

Generally a ground voltage level is set to negate noise, where the first signal in the envelope is the first to break the threshold. Additional parameters are determined from the wave signal using this method, such as average frequency and RA value. RA value is the rise time divided by the amplitude, which can be used to classify the type of crack. [30] Although this method is quick and efficient, further insight into the AE wave properties can provide more information about the source and the substrate. A more advanced analysis technique is needed to extract this information, which can be provided by waveform analysis.
B. Waveform Analysis

Waveform analysis is the more modern form of investigating acoustic emission signals. It has been developed with advancing computing power, since the entire signal from the sensor is recorded and analyzed. A facet of waveform analysis, modal analysis, can be used to verify the sensitivity of a sensor to various components of an AE wave. Most systems have an inherent noise that must be separated from the usable AE signal, and modal analysis provides some insight into the frequency characteristics of the signal. Understanding the frequency characteristics of the stress waves in the substrate allows for effective signal processing, e.g. band-pass filtration and spectral analysis. The frequency characteristics of AE waves are dependent on several parameters, such as the substrate material and geometry and sensor geometry. For example, the experiments conducted for this thesis were conducted on a 309 stainless steel thin bar substrate, with cross section dimensions of 6.50 mm thick and 26.7 mm wide and a length of 913 mm. Thus Lamb waves are expected for the thin substrate over Rayleigh or Love waves, since Lamb waves occur when the substrate takes a plate-like form, generally thinner than or comparable to the wavelength of the propagating waveform. An illustration of the substrate and supports is shown in Figure 4.
Lamb waves take a complex form that is a combination of extensional and flexural waves [31]. These wave modes are referred to as symmetric for extensional, and antisymmetric for flexural, due to the theoretical shape that the substrate takes when the wave propagates. An illustration of this concept is shown in Figure 5.
The dispersion curve reveals the relationship between the group or phase velocity and frequency for each wave mode, thus, the dispersion curve for the test substrate was analyzed to verify Lamb wave modes in the sensor signal obtained in experiments. The relationship between each layer of the substrate, including the atmosphere on top and bottom, can be modeled using the global matrix for a particular substrate, which is used to calculate the dispersion curve. This global matrix is represented by $G$, shown in Equation (1) [31].

$$
G = \begin{bmatrix}
D_{1b} & D_{2t} & D_{3t} & \ldots \\
D_{2b} & D_{3t} & D_{3b} & \ldots \\
& \ddots & \ddots & \ddots \\
& & D_{(n-1)t} & D_{(n-1)b} & D_{nht}
\end{bmatrix}
$$
In the global matrix, each $D_{ij}$ term represents the transmission or reflection properties between each layer, and is a function of the wave number, frequency, wave velocity, layer density, and layer thickness. The element $D_{it}$ represents these properties for the top surface of a layer, and $D_{ib}$ represents the same for the bottom surface. The first and last terms, $D_{1hb}$ and $D_{nht}$, are for the bottom half-space layer and the top half-space layer, which are the first and last layers for energy dissipation. By setting the determinant of the global matrix equal to zero, and solving for each mode’s frequency as a function of velocity by iteration, the dispersion curve can be created [31].

As an example, the dispersion curves for stainless steel 309 are shown below. PACshare Dispersion Curves commercial freeware was used to create the dispersion curve. The dispersion curve for the single layer 309 stainless steel substrate is shown in Figure 6, and the parameters used for the calculation are shown in Table 3. The wave velocity is the same as group velocity for this application. The labels in the figure mark the antisymmetric modes with the letter “A” and the symmetric modes with the letter “S”, accompanied by wave order, with zero, one, and two shown.
Table 3: Dispersion curve calculation parameters

<table>
<thead>
<tr>
<th>Layer</th>
<th>Thickness</th>
<th>Longitudinal Velocity</th>
<th>Shear Velocity</th>
<th>Surface Velocity</th>
<th>Acoustic Impedance</th>
<th>Density (kg/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>$\infty$</td>
<td>0.34 km/s</td>
<td>N/A</td>
<td>N/A</td>
<td>411 Rayl</td>
<td>1.12 kg/m$^3$</td>
</tr>
<tr>
<td>309 SS</td>
<td>6.5 mm</td>
<td>5 km/s</td>
<td>3.102 km/s</td>
<td>2.78 km/s</td>
<td>45 MRayl</td>
<td>8000 kg/m$^3$</td>
</tr>
<tr>
<td>Air</td>
<td>$\infty$</td>
<td>0.34 km/s</td>
<td>N/A</td>
<td>N/A</td>
<td>411 Rayl</td>
<td>1.12 kg/m$^3$</td>
</tr>
</tbody>
</table>

The regions where the mode lines are vertical show the frequencies where the modes will appear in the frequency spectrum. For each of the modes above the zero order, this corresponds to their nascent frequency, which is the minimum frequency at which those modes will appear. The dispersion curve can also be used to show how the wave mode frequency changes with time. The velocity axis can be converted to time as long as the
source distance is known and does not change. The dispersion curve relating time and frequency is shown in Figure 7, with a source distance of 200 mm used for this calculation.

For example, using figure 6 and 7, the zero and first order antisymmetric modes can be expected at a very low frequency and approximately 250 kHz, respectively. The frequency associated with the zero order symmetric mode is more difficult to predict, but the time of arrival should be approximately 20 microseconds early than that of the zero order antisymmetric mode.

![Dispersion Curve (Known Source)](image)

Figure 7: Dispersion curve for a source distance of 200 mm.

After the AE sensor data is processed in the time and frequency domain, a spectrogram of the results can be created [32]. The spectrogram relates the frequency component of the data with time. By overlaying the theoretical dispersion curve and
empirical spectrogram, the influence of each wave mode can be seen, and the ability for a sensor to detect various wave modes can be assessed.

This form of post-processing analysis is used in this paper to evaluate the wave modes in the time and frequency domains, and to gauge the ability of the YCOB sensor to detect the distinct wave modes, which is critical for an acoustic emission sensor to be feasible for structural health monitoring.

C. FEA Modeling

The frequency components of the Lamb wave that will be detected by the sensor are difficult to predict using an analytical model, so finite element analysis was conducted to help verify the characteristics of the signal received from our sensor. The FEA program COMSOL was used for modeling. The Hsu-Nielsen AE source is modeled as a uniformly distributed ramp load with a rise time of 0.5 µs and peak amplitude of 3 kNm$^{-2}$ for an area of 0.4 mm$^2$[33, 34]. This is a much simpler source to model than fatigue cracking, so it was used to simulate the Lamb waves. With known substrate properties, an AE wave can be closely modeled and the wave characteristics can be determined with the appropriate FEA probes [35]. The parameters used to model the AE wave are shown in Table 4.
## Table 4: Parameters for finite element modeling.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bar model dimensions</td>
<td>400 mm x 26.7 mm x 6.5 mm</td>
</tr>
<tr>
<td>Plate model dimensions</td>
<td>200 mm x 200 mm x 6.5 mm</td>
</tr>
<tr>
<td>Substrate Density</td>
<td>8000 kg/m³</td>
</tr>
<tr>
<td>Substrate Young’s Modulus</td>
<td>200 GPa</td>
</tr>
<tr>
<td>Substrate Shear Modulus</td>
<td>77 GPa</td>
</tr>
<tr>
<td>Mesh size maximum</td>
<td>0.5 mm</td>
</tr>
<tr>
<td>Mesh size minimum</td>
<td>1 µm</td>
</tr>
<tr>
<td>Time range</td>
<td>50 µs</td>
</tr>
<tr>
<td>Time step size</td>
<td>1 µs</td>
</tr>
<tr>
<td>Probe distance from force</td>
<td>50 mm</td>
</tr>
</tbody>
</table>

Smaller dimensions and a shorter time range were used to reduce the calculation time. The wave modes are only dependent on the thickness and material properties, so the outer dimensions and probe distance do not affect the stress wave frequency characteristics. An out-of-plane surface displacement probe was used since there should be little frequency change between the plate wave and the signal produced by the piezoelectric sensor. The sampling rate and mesh size were chosen to allow for distinct wave frequencies at the expected antisymmetric and symmetric nascent frequencies. The properties of 309 stainless steel were used to define the substrate material, and the force was induced at the center of the model with the parameters given at the beginning of this chapter. A multiphysics package was not used; since we were only deciphering the frequency characteristics of the wave, only a solid mechanics package was necessary.

The finite element analysis was also conducted to analyze the cause of unexpected behavior in the frequency and spectrogram plots in Chapter 6. The double band that is seen at
30 kHz and 70 kHz in the room temperature and high temperature spectrograms, in Chapter 6.1, was not expected. Based on the dispersion curve calculated, the 30 kHz frequency peak is anticipated, but a second peak in close proximity, like the one at 70 kHz, is not usually prevalent in acoustic emission testing. This is due to the fact that the test was conducted on a bar, whereas acoustic emission tests are usually conducted on plates, which are large and thin enough to ignore reflections in the early sensor response. To verify this, a bar and a plate substrate were both modeled. The frequency domain of the modeled response on the bar and plate are shown in Figure 8.
The peak at 70 kHz is clearly not prevalent in the full plate model, and can be attributed to the boundaries created on the sides of the bar. The shift that is seen in the A1 mode is unexpected, but explains the double spikes in the frequency components at 250 kHz and 300 kHz, as shown in Figure 17.

Figure 8: FEA results for bar (a) and plate (b) - frequency domain.
IV. SENSOR DESIGN AND EXPERIMENTAL METHODS

A. Sensor Design

For the sensor to properly transmit the AE waves created experimentally, the design and orientation was carefully considered. Acoustic emission sensors and accelerometers usually take advantage of one of two configurations, either a compressive or a shear style that is dependent on the properties of the piezoelectric material. Compressive sensors typically use a wear plate to separate the piezoelectric material from the substrate, but otherwise put the sensing element as mechanical close to the substrate as possible, using bonding agents or mechanical clamping. Shear mode piezoelectric sensors sandwich the sensing element between the body of the sensor and a seismic mass in an orientation normal to the substrate, creating shear stress. An illustration of both sensor styles is shown in Figure 9, with exaggerated sensing element and mass sizes for clarity.
The shear style sensor would be necessary for this experiment due to the properties of YCOB. The YCOB single crystal used in this experiment was cut to take advantage of its highest thickness piezoelectric constant, shear mode $d_{26}$. This thickness shear mode is illustrated in Figure 10, where the dashed lines show the original shape, the solid lines show the shape after stress is applied, and the arrows show the direction of stress and voltage output.
The piezoelectric constants for YCOB are shown in Table 5, from Shimizu et al. [36] and communications from the Materials Research Institute at Pennsylvania State University [37].

Table 5: Piezoelectric properties of YCOB.

<table>
<thead>
<tr>
<th>Source</th>
<th>$d_{11}$</th>
<th>$d_{12}$</th>
<th>$d_{13}$</th>
<th>$d_{15}$</th>
<th>$d_{24}$</th>
<th>$d_{26}$</th>
<th>$d_{31}$</th>
<th>$d_{32}$</th>
<th>$d_{33}$</th>
<th>$d_{35}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shimizu et al.</td>
<td>1.4</td>
<td>3.8</td>
<td>-4.2</td>
<td>-7.2</td>
<td>-2.6</td>
<td>8</td>
<td>-0.22</td>
<td>-2.3</td>
<td>0.83</td>
<td>2.2</td>
</tr>
<tr>
<td>MSI at PSU</td>
<td>1.7</td>
<td>--</td>
<td>--</td>
<td>-1.1</td>
<td>4.4</td>
<td>--</td>
<td>-0.77</td>
<td>--</td>
<td>1.4</td>
<td>-5.0</td>
</tr>
</tbody>
</table>

Some reported high temperature acoustic emission sensors were tested by heating the substrate that they rest on, but this method can only be used for a limited range of temperatures [21]. For this reason, the sensor was tested inside of a tube furnace. The furnace used had an inner diameter of less than 2 inches, so the substrate, sensor, and any clamping device were designed to fit within this small space. Due to this space restriction the seismic...
mass style shear sensor is not feasible for this experiment. Bonding agents are also not usable at the testing temperatures. Even high temperature bonding epoxies crack and degrade at 1000 °C, which would limit the sensor’s ability to detect stress waves. Instead of using vertically oriented piezoelectric layers, the crystal was applied directly to the substrate surface, and clamped in place using a small diameter c-clamp. The clamp can withstand the necessary high temperatures, and it created a strain difference between the top and bottom of the crystal, thus transmitting shear stress. Although this could be a potential cause of reduced sensitivity, it was necessary in order to fit the sensor with a high temperature furnace, and did not critically inhibit the device.

The rest of the AE sensor was constructed using materials that could withstand the 1000 °C maximum temperatures. Based on material properties and the design of the YCOB accelerometer fabricated by K. Kim et al [25], Inconel was chosen as the electrode and wire material. The bottom electrode was removed to reduce the number of layers the stress wave must travel through, increasing the sensitivity. The YCOB plate with dimension of 8 mm × 6mm × 2mm was in contact with the 309 stainless steel substrate, which thus acted as the bottom electrode in the sensor.

The sensor consisted of a simple stack with only a top electrode, an insulator pad, the piezoelectric crystal, and the clamp, as shown in Figure 11. A 316 stainless steel clamp was used, which has a critical operating temperature at around 1000 °C, but can be used for short time intervals. The insulator pad was used to electrically isolate the top electrode from the
clamp. A photo of the sensor is shown in Figure 12. This sensor was also used for the room temperature testing to ensure consistent frequency and amplitude wave characteristics.

Figure 11: High temperature sensor design.

Figure 12: High temperature sensor photo.
B. Experimental methods

There are many methods for determining the effectiveness of an acoustic emission sensor. The methods can generally be described as either simplified reproductions of applications that produce real AE stress waves, or designed to replicate AE sources that are otherwise found in industrial environments. Replications of real applications include tests such as induced fatigue or corrosion cracking, and will give a more accurate signal but require more equipment and may create more system noise that will need to be removed. On the other hand, methods designed to imitate real AE sources are easier to reproduce and isolate from noise sources, are cheaper, but create artificial signals that may not show signal characteristics that would show genuine applications. Therefore, in this thesis, both methods are used. A cheap method designed to reproduce AE induced Lamb waves is initially used to verify the ability of the YCOB sensor to detect the wave modes, and then a fatigue crack test is used to verify that the sensor could detect more accurate stress waves, all at high temperatures.

The most widely used acoustic emission in sensor research is the Hsu-Nielsen test, in which a reproducible acoustic emission source creates a stress wave very similar to actual sources, such as crack propagation. The Hsu-Nielsen source is a pencil lead break on the surface of the test object. The most common type of lead used is 0.5mm with a hardness of 2H. The lead is held using a lead holder, or mechanical pencil in most cases, and is inserted through a plastic “boot”, as shown in Figure 12, used to reduce stress wave interference from vibration. The boot is also intended to hold the lead at a constant angle for each test [38].
This method was used to test the sensitivity of YCOB and compare it to a high performance PMN-PT crystal.

In order to test the YCOB sensor against a more realistic wave source, a fatigue crack experiment was used. Although fatigue crack tests can vary widely, a bending cantilever beam with a notch was used for this thesis. The same substrate was used for comparable Lamb waves, and the beam was vibrated using a shaker until fatigue cracking occurred within the notch.

C. Hsu Nielsen experimental design and setup

The preliminary testing for sensitivity was conducted by performing the Hsu-Nielsen test with the source 20 cm away from the PMN-PT and YCOB AE sensors. The signal from the piezoelectric crystal was amplified using a Brüel & Kjær Charge Amplifier Type 2635, received and recorded using an Agilent Technologies InfiniiVision DSO7104B oscilloscope. The data was transferred to a computer with MATLAB, PACshare Dispersion Curves, and Vallen Wavelet software for signal processing and analysis. The code for all processing was written in MATLAB.

The sensor tested in this research was located in the center of a horizontally oriented tube furnace. The pencil lead was broken outside of the insulated section of the tube furnace, and the Inconel wire extended out of the high temperature region to connect to the amplifier. A-frame stands and clamps were used to hold the substrate in place.

The furnace (MTI Corporation GSL1100X) was heated by 100 °C increments up to 1000 °C. The furnace would be heated to a value above the target temperature, and then
unplugged to reduce the AC electric noise created by the heating units and allowed to cool during testing. This allowed for an average testing temperature at the target temperature. The Hsu-Nielsen setup is shown in Figure 13.

Figure 13: High temperature Hsu-Nielsen acoustic emission testing setup.

This testing setup allowed for repeatable lamb waves generated from the Hsu-Nielsen source, with limited change in the signal characteristics. These small changes in the signal are further discussed in Chapter 6.

D. Fatigue cracking experimental design and setup

The fatigue cracking experiment took advantage of the same furnace, substrate, test stand, sensor, charge amplifier and oscilloscope. In addition to this equipment, a Tektronix
AFG3101 function generator was used to create a driving signal for a Vibration Test Systems VG-100-8 Shaker, with the signal amplified using a Kepco bipolar operational power supply. The shaker was attached to one side of the stainless steel bar substrate, with the other end clamped. This created a cantilever beam substrate. A notch was cut into the substrate in order to induce a fatigue crack prior to the high temperature tests. An illustration of this test setup is shown in Figure 14.

![Figure 14: High temperature fatigue cracking acoustic emission testing setup.](image)

The cantilever beam was bent until a crack was induced. The notch was watched closely, and vibration was stopped once a crack was produced so that ample data could be taken before the crack propagated through the bar and caused it to break. To predict the
necessary force created by the shaker, the stress required to produce a crack was calculated. A PCB accelerometer was used to measure the displacement at the tip of the beam in order to relate the voltage amplitude output from the function generator to the force produced by the shaker. The necessary stress was calculated using Equations (2) and (3) [39].

\[
\sigma = \frac{K_I}{\sqrt{\pi a}} \frac{2b}{\pi a \tan \left( \frac{\pi a}{2b} \right) G_I}
\]

(2)

Where:

\[
G_I = \frac{0.923 + 0.199 \left( 1 - \sin \left( \frac{\pi a}{2b} \right) \right)^4}{\cos \left( \frac{\pi a}{2b} \right)}
\]

(3)

The value \( K_I \) is a property of geometry, referred to as the stress intensity factor. When it equals the fracture toughness of a material, theoretically, a crack will propagate. For fatigue cracking testing, the full \( K_I \) value does not need to be met, only approximately half of it. The values \( a \) and \( b \) represent the notch length and width of substrate. Since this experiment used a bending beam orientation, the value \( b \) corresponded to the thickness of the substrate since the notch was cut into the thickness. An illustration of the crack dimensions and stress orientation is shown in Figure 15.
To relate the necessary stress to an input force at the end of the beam, Equation (4) was used, which is a commonly known relationship in beam bending:

\[ \sigma = \frac{My}{I} = \frac{FLb}{2I} \]  \hspace{1cm} (4)

where F is the tip force, L is the length to the point of interest (the notch), and I is the moment of inertia. For this bending beam, the notch changes the actual stress from what this simplified equation presents. For these basic calculations, the thickness was assumed to be equal to the thickness b minus the notch length a. Equation (4) normally only holds true for static loads, but since the cyclic load applied in this experiment was of very low frequency the assumption was made that vibrating wave modes would not be necessary for consideration. Since an accelerometer was being used for calibration, equations relating the force to deflection, and deflection to acceleration are likewise needed. These relationships are shown in Equations (5) and (6), respectively.

\[ \delta = \frac{FL^3}{3EI} \]  \hspace{1cm} (5)

\[ \delta = \frac{A}{\omega^2} \]  \hspace{1cm} (6)

Figure 15: Notch dimensions and stress.
Where $A$ is the acceleration measured at the tip, and $\omega$ is the frequency. These five equations were used to estimate when the cracking would occur. The values assumed for this test are given in Table 6.

Table 6: Parameters for crack inducing stress calculations.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bar thickness</td>
<td>6.5 mm</td>
</tr>
<tr>
<td>Notch Length</td>
<td>3.2 mm</td>
</tr>
<tr>
<td>Length to Crack</td>
<td>430 mm</td>
</tr>
<tr>
<td>Moment of Inertia</td>
<td>606.5 mm$^4$</td>
</tr>
<tr>
<td>Substrate Fracture Toughness</td>
<td>100 MN/m$^{3/2}$</td>
</tr>
<tr>
<td>Substrate Young’s Modulus</td>
<td>200 GPa</td>
</tr>
<tr>
<td>Input Frequency</td>
<td>10 Hz</td>
</tr>
</tbody>
</table>

Although these calculations do not give an exact necessary input voltage, they did allow for ballpark calculations that provided cracking within a reasonable time period. This fatigue cracking test provided realistic acoustic emission stress waves with which to test the YCOB sensor.
V. RESULTS AND DISCUSSION

A. Hsu-Nielsen Experimental Results

The initial analysis of the Hsu-Nielsen test at room temperature was conducted purely in the time domain, by comparing the response of the PMN-PT and YCOB AE sensors. Due to the differences in the magnitude of the piezoelectric constants of the two crystals (~8pC/N for YCOB versus >1500pC/N for PMN-PT), a higher amplification is needed for the YCOB AE signal. For testing at room temperature using the experimental setup in Figure 12, the YCOB AE sensor signal was amplified by 100 times with the charge amplifier, and the PMN-PT AE sensor signal was not amplified. For a Hsu-Nielsen test from 20 cm, typical time responses for both crystals are shown in Figure 16.
Although the time windows are different for the two AE sensors, the high frequency and low frequency components of the wave are both apparent in the received YCOB and PMN-PT AE signals. The symmetric and antisymmetric components can be recognized with frequency spectrum analysis. The time domain signal was analyzed in the frequency domain by performing a Fast Fourier Transform, and finding the power spectral density (PSD). The frequency responses for both signals are shown in Figure 17. One can clearly see the

Figure 16: Room temperature Hsu-Nielsen time-domain responses
dominance of the symmetric and antisymmetric modes in terms of the spectral density, particularly in the case of the YCOB crystal.

Figure 17: Frequency responses at room temperature of YCOB and PMN-PT.

The antisymmetric and symmetric zero order modes can be discerned in the frequency spectrum of the signals produced by the AE sensor from the Hsu-Nielsen and fatigue crack test, but relating these frequencies to the dispersion curves for the substrate
requires another set of tools. The modes that are pointed out in Figures 17, 20 and 24 can be verified using a combination of dispersion curve and spectrogram software. The spectrogram software shows how the frequency of the signal changes with time, exposing the separate modes. The combination of these two for the YCOB AE sensor response to the Hsu-Nielsen source at room temperature is shown in Figure 18.

![YCOB Room Temperature Spectrogram](image)

Figure 18: Spectrogram of YCOB sensor voltage at room temperature.

Spectrograms can show distinct frequencies when applied to ultrasonic signals, but the complex modes of Lamb waves in acoustic emission signals do not appear as well-defined lines [32,40]. The frequency components of the spectrogram follow the dispersion curve lines, which are corrected for the source distance and any time bias from moving the
time window of the original signal. The influence of the A0 and S0 modes is distinguishable in Figure 18, but the A1 mode is less prevalent. The A1 250 kHz frequency range component can be seen in the frequency domain plot for both YCOB and PMN-PT in response to the Hsu-Nielsen source. These frequencies can be seen at all temperature levels, with some shifting as the temperature increases. This can be expected due to the changing mechanical properties of the steel bar with the increasing temperature. The double bands at 30 and 70 kHz are due to the geometry of the substrate, which is verified in section 3.3. The dispersion curve is calculated for a semi-infinite plate, but the stainless steel bar substrate has a width on the same scale as the thickness, approximately four times the thickness. As previously discussed, this boundary influences the harmonics of the Lamb wave frequencies.

The spectrogram can also be verified through time of arrival [40]. In Figure 16, the center of the S0 mode appears at a time of approximately 45 µs, and the center of the A0 region appears at approximately 80 µs. Due to the manual nature of the testing, time of arrival difference is a better verification method than individual time of arrival since there is no reference time of zero seconds. The group velocity of the A0 mode at 30 kHz is approximately 2800 m/s, and the group velocity of the S0 mode at 120 kHz is approximately 4800 m/s. These velocities are taken from Figure 6, and the frequencies can be taken from Figure 17, 20, or 24. Equation (7) is used to find the time of arrival difference for the two modes, with a known source distance.
\[
\Delta t = t_A - t_S = \frac{d}{V_A} - \frac{d}{V_S} = \frac{d(V_S - V_A)}{V_S V_A}
\]  

(7)

For a distance of 20 cm and previously stated velocities, the time arrival difference should be about 38 µs, which is in close agreement with the experimental results in Figure 18.

The ability to detect the Lamb wave modes displayed by YCOB AE sensor is very promising for acoustic emission sensor applications. Any handicap that the YCOB crystal may have due to its lower piezoelectric constant is reduced with the ability to discern separate modes within its signal. The voltage response of the YCOB AE sensor to the Hsu-Nielsen test at 1000 °C is shown in Figure 19, with a room temperature signal for comparison. The magnitude differences between the signals are due to a reduced amplification necessary to record the signal with increased noise. After factoring out amplification, the magnitudes are on the same scale.
The increased noise levels are apparent, but the high frequency and low frequency components of the original signal are still distinct. The same FFT and PSD were applied to this high temperature response in order to analyze the frequency characteristics. Although the frequency peaks are not as dominant in the Fourier Transform, the zero order modes are still the most prevalent and higher order modes are visible. The frequency response received from the YCOB AE sensor at 1000 °C is shown in Figure 20.

Figure 19: Hsu-Nielsen AE signals at room temperature and 1000 °C.
The spectrogram and dispersion plot for the YCOB AE sensor voltage output in response to the Hsu-Nielsen test at 1000 °C are shown in Figure 21. The results are comparable to the graph at room temperature, with clear influence from the S0 and A0 modes, and little influence from the A1 mode. The region with increased amplitude at a time of 170 µs is due to reflection, since the high temperature test used clamps instead of simple supports for the substrate.
B. Fatigue cracking test results

The capability of the YCOB sensor to detect fatigue cracking at high temperatures was examined. The signal from the YCOB sensor showing the vibration is displayed in Figure 22. The 100 millisecond pattern shown corresponds to the 10 Hz shaker frequency. The fatigue cracking test produced sensor outputs that primarily displayed the stress produced by the shaker, which can be seen by a large magnitude voltage every 100 ms. Over a large time range, where the vibration was apparent, the AE signal could not be easily distinguished. In order to detect these high frequency components it was necessary to examine the signal over a smaller time domain. The voltage produced by the sensor was amplified by a factor of 10.

Figure 21: Spectrogram of YCOB voltage output at 1000 °C.
The high frequency signals are seen by locking the signal and decreasing the time window around dense regions of the signal. Ideally the entire signal would be recorded at a very sampling rate, but the oscilloscope could not record an entire vibration period with enough samples to discern Lamb waves. Therefore the high frequency acoustic emission signals were located within the locked signal and short time period signals were recorded. An acoustic emission signal produced by the fatigue crack at 1000°C is shown in Figure 22.

Figure 22: Vibration signal at 1000 °C.
The frequency components of this acoustic emission signal can likewise be analyzed, as shown in Figure 24, using methods previously described in this work.
The zero order antisymmetric mode has a very strong presence in the sensor signal, but the symmetric mode peak is only approximately 10 dB above the noise floor around it. This is likely due to the difference in stress wave frequency released by the fatigue crack, compared to the Hsu-Nielsen source. As discussed in Chapter 1.1 and shown in Figure 2, the frequency component of the AE signal is dependent on the source. The frequency of the fatigue crack signal likely excites lower frequency Lamb wave modes, whereas the Hsu-Nielsen source excites higher order modes. Crack propagation events, like the ones created by the Hsu-Nielsen source, can create signals with higher frequency components when compared to fatigue cracking. This comparison is shown in Figure 2. The dispersion curve and spectrogram plot for the fatigue crack test is not shown because only the zero order antisymmetric modes are prevalent, so the spectrogram only shows the horizontal line at the bottom and does not offer useful information.

C. Discussion

Besides the frequency characteristics, the voltage amplitude output from the YCOB crystal gives a good measure of sensitivity. The peak to peak voltage was recorded for the Hsu-Nielsen test, and the mean and standard deviation at each temperature level are shown in Figure 25. The peak to peak voltage is not shown for the fatigue crack test because this stress wave is a function of the crack movement, and varied with each oscillation.
The average value for peak to peak voltage across all temperature levels was 0.06248 V. From Figure 25, it can be seen that there is no major relationship between peak to peak voltage and temperature. This can be expected in reflection of recent publications on YCOB’s stability at high temperatures, such as Tittmann’s bake test and Zhang’s testing of high temperature ReCa₄O(BO₃)₃ materials, which includes YCOB [26, 41].

There is certainly room for improvement and necessary future research for the development on this high temperature acoustic emission sensor. Ideally the sensor needs to be incorporated into a housing, similar to the high temperature acoustic emission sensor in Table 2 that has an Inconel housing. This helps to reduce noise and protect the critical sensor parts from physical damage. Likewise, the Inconel electrode and wire used were very crude. A wire with a better contact, and possibly better material all together could be used. During
the sensor assembly, the electrode, insulator and YCOB crystal were compressed by hand using the clamp. A method of applied a measured clamping pressure could be incorporated into a sensor housing. This would prevent overly compressing and damaging the YCOB crystal or the ceramic alumina insulation layer, and allowing for maximum transmission of the shear stress wave.

Improvements on testing equipment could also advance the signal quality by being able to record entire vibration signals during fatigue crack testing. Filters would eliminate the vibration signal and corresponding harmonics, theoretically leaving only the high frequency acoustic emission signals and background noise over a long time period. Taking multiple acoustic emission signals within one sample allows for the possibility of more advanced frequency analysis.

For application in nuclear environments, testing on the effect of radiation on the sensor would be necessary. With a nuclear reactor on campus at North Carolina State University, this is not an infeasible experiment. Long-term effects, as well as short-time testing similar to the testing conducted in this thesis would be necessary. Introduction of the sensor into combustion environments would require research on the effect of high pressure on the sensor as well. While piezoelectric materials are commonly used for pressure sensors, in this case the stress induced by the pressure would need to be minimized or filtered out of the resulting signal.
With further research and development of a high temperature YCOB acoustic emission sensor, structural health monitoring in extreme environments, including nuclear and combustion systems, is within reach.
VI. CONCLUSION

YCOB acoustic emission sensor was designed, fabricated and tested, demonstrating the capability of detecting acoustic emission stress waves at temperatures up to 1000 °C. Both zero order lamb wave modes at frequency of 30 kHz and 120 kHz and the first order antisymmetric wave mode at frequency of 240 kHz can be detected by the YCOB AE sensor during Hsu-Nielsen testing. The AE signal could be picked out during fatigue crack testing apart from the low frequency vibration, but only the antisymmetric Lamb wave mode made a strong presence. This could be due to the stress source, or due to the additional noise created by the vibration. The frequency characteristics of the detected waves were verified using the FEA software COMSOL and spectrogram analysis. For most applications, an acoustic emission sensor is expected to at least detect the A0 and S0 mode. Both of these modes can be seen in the frequency domain for both Hsu-Nielsen and fatigue crack test; thus the YCOB AE sensor is viable for acoustic emission detection with signal frequency component analysis. With further development in sensor design and signal analysis, as well as research on the sensor’s reaction other extreme environment conditions such as radiation and high pressure, structural health monitoring in extreme environments using a YCOB acoustic emission sensor could be implemented.
REFERENCES


