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Report

ELEVATED TEMPERATURE SENSORS FOR ON-LINE CRITICAL EQUIPMENT HEALTH MONITORING

March 2005

Elevated Temperature Sensors for On-Line Critical Equipment Health Monitoring

Annual Report

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ABSTRACT

The objective of this research program is to improve high temperature piezoelectric aluminum nitride (AlN) sensor technology to make it useful for instrumentation and health monitoring of current and future electrical power generation equipment. The program will extend the temperature range of the sensor from approximately 700°C to above 1000°C, and ultrasonic coupling to objects at these temperatures will be investigated and tailored for use with the sensor. The chemical vapor deposition (CVD) AlN deposition process was successfully transferred from film production on tungsten carbide substrates to titanium alloy and silicon carbide (SiC) substrates in the first year of the program, and additional substrates were evaluated. In the second year of the program, additional substrate research was performed with the goal of improving the performance of using SiC substrates. While greatly improved bandwidth was achieved, sensor survival at elevated temperature remains problematic. The elevated temperature coupling work continued with significant experimentation. Molten glasses were found to work within a limited temperature range, but metal foils applied with heat and pressure were found to have superior performance overall. The final year of the program will be dedicated to making further advances in AlN/ substrate behavior, and the design and implementation of a sensor demonstration experiment at very high temperature in a simulated industrial application.

TABLE OF CONTENTS

Section | **Page**

SECTION 1
INTRODUCTION

In order to use coal-fired electrical power generation equipment to its fullest potential, sensors are required which are capable of monitoring the "health" of critical system components. Such sensors can alert an operator to impending problems, if any, while reducing the need to shut down equipment for inspection. Benefits of health monitoring include reduced down time for inspection or unplanned outages. For equipment operating at high temperatures, this will also likely reduce thermal cycles and associated fatigue or wear. Equipment that can benefit from such health monitoring includes dynamic systems, such as turbine engines, as well as static systems such as gasifiers, process piping, or other chemical processing facilities.

Past research conducted at the University of Dayton into the properties and behavior of Aluminum Nitride (AlN), a high temperature piezoelectric material, indicated that this material has a high potential for providing a basis for sensors that can be used for elevated temperature health monitoring, up to at least 1000°C (1800°F). The most straightforward implementation of such AlN sensors is as ultrasonic transducers, for example as a thickness monitor in a critical region of a metal or ceramic component exposed to corrosive or erosive conditions. Ultrasonic thickness gages are widely available commercially for room temperature measurements. Ultrasonic waves may additionally be used to monitor bonds, such as that between a metal and a ceramic, as found in thermal and environmental barrier coatings commonly found in power generation equipment.

The first year of this three-year effort focused on laying the groundwork for extending the sensor technology to the high temperature (>1000°C) regime. The second year, described in this document, continued to extend this groundwork. The first-year investigations of high temperature ultrasonic coupling were reduced to practice and were evaluated experimentally. Additional development work on substrate materials confirmed the desirability of switching from tungsten carbide to silicon carbide substrates, and progress was made toward making this switch. The design of the sensor surrounding the active element progressed, including evaluation of suitable hot-section cabling, the overall housing, and the conceptual design of a low-cost circuit that could be used to drive the sensor and condition the ultrasonic signals into usable data.

SECTION 2
EXECUTIVE SUMMARY

The goal of this project is to develop sensors that can be used at elevated temperatures, up to 1000°C, in power generation equipment. The primary target application for these sensors is equipment health monitoring. Historically, ultrasonic sensors have been used to evaluate properties of solid and fluid materials. Detected flaws may include cracks, voids, inclusions, porosity, or other discontinuities. Additionally, ultrasound is used in flowmeters, proximity sensors, and for material thickness measurements.

Conventional ultrasonic transducers and instrumentation are temperature limited because the piezoelectric materials used in the transducer cannot operate above their Curie point or de-poling temperature. Operation above 100°C is considered "high temperature" with special requirements met by quartz or tourmaline transducers up to ~450°C, or complicated delay lines with complex cooling requirements (and usually) exposure times measured in seconds. Laser-based ultrasound and electromagnetic acoustic transducers (EMATs) are also used at high temperatures but are expensive or complicated. In addition, coupling the ultrasonic energy from the transducer into the test object becomes much more difficult above 100°C.

In the current project, ultrasonic transducers based on piezoelectric aluminum nitride (AlN) films are being developed which will enable the use of ultrasound for material evaluation at temperatures exceeding 1000°C. The preexisting process for film development produced films on tungsten carbide substrates, and an important part of the current project is to develop films on another substrate capable of improved operation at the 1000° target temperature. The resulting sensor will be coupled into a test object for demonstration at a very high temperature, the goal being to operate above 1000°C.

The first year of the program saw titanium metal attempted and discarded as a candidate material. In the second year, various grades of silicon carbide were attempted with results superior to tungsten carbide but not yet suitable for a robust sensor. Signal strength of the SiC substrate transducers equaled those on tungsten carbide, and significantly better bandwidths were achieved. An informal partnership with an advanced ceramics group of Saint-Gobain is developing which is expected to result in the selection of a silicon carbide that will be used in the demonstration sensor experiment at the end of the program. In addition, a sample of a novel, electrically conductive aluminum nitride was procured and a film was successfully deposited on it. Additional materials were considered and discarded, primarily due to thermal expansion concerns after the first year's experience with titanium.

The results from the first year's literature survey on elevated temperature ultrasonic coupling pointed toward the use of molten glass or metal foil as a couplant material. These methods were further investigated and demonstrated experimentally in the second year. The use of molten glass was touchy; it worked but tended to react with materials in contact with it, as well as the air. Metal foils showed much more promise as a robust couplant, beginning with aluminum foil at lower temperatures and extending on to gold and possibly platinum at higher temperatures. In the final year of the program, the foil coupling and improved film/substrate sensor core will be integrated into a sensor for a demonstration experiment.

SECTION 3
EXPERIMENTAL

Piezoelectric AlN Film Deposition

The basic process for film deposition was described in the report for the previous year. In the current year, the same CVD process was used to attempt to transition the deposition onto silicon carbide (SiC) substrates, rather than the cobalt-bonded tungsten carbide (WC) cermet that had historically been the substrate of choice. Two primary factors motivated this transition:

- The SiC is a better selection for use at temperatures above 700°C. A coherent, protective silicon oxide forms at the surface, which protects it against further oxidation to temperatures beyond 1000°C. Use of SiC would ensure that the AlN sensor material's upper range is not limited by a substrate that oxidizes before it does. (AlN becomes sensitive to oxidation in the vicinity of 800°C.)
- SiC is a much better acoustic impedance match for AlN. This is necessary to create a high bandwidth ultrasonic sensor capable of producing a pulse with minimal ringing. The difference between WC and SiC is shown in Figures 1 and 2. A high bandwidth signal provides better time resolution and sensor performance.

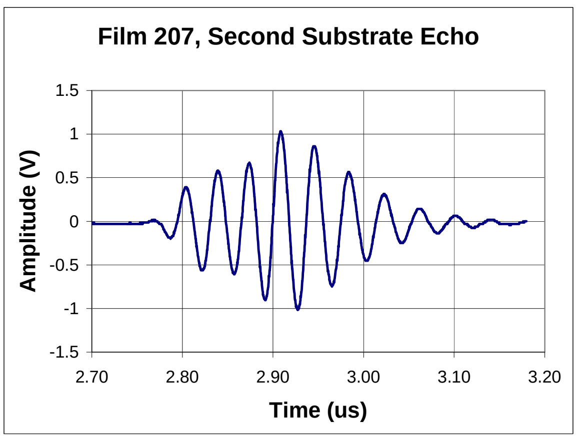

Figure 1. Narrowband ultrasonic signal with significant ringing produced by AlN film on WC substrate.

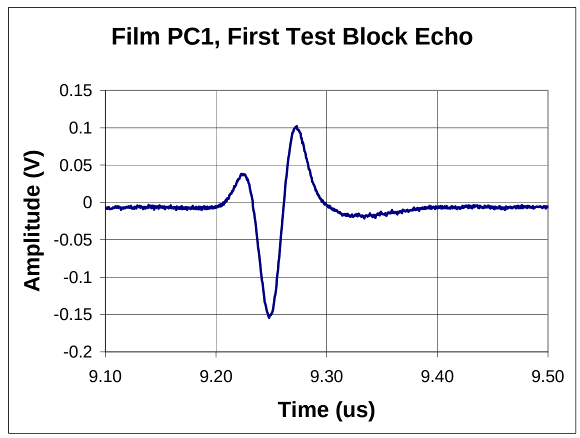

Figure 2. Wideband ultrasonic signal produced by AlN film on SiC substrate

SiC shapes are available in a number of different material sub-types, classified by composition and production method. Direct-sintered SiC is prepared by sintering SiC particles under heat and pressure to form a solid material. Reaction-bonded SiC is prepared from a blend of SiC and carbon powders, green pressed and infiltrated with molten silicon which reacts with the carbon, generally leaving some free silicon in the finished part. Solid SiC shapes may also be prepared by laying down successive layers with a chemical vapor deposition process, the most expensive method. Since the substrate is used as one of the electrodes to apply voltage across the AlN film, the most conductive SiC materials were sought out for AlN deposition trials, with reaction-bonded excluded from the trials because of the free silicon.

Initial deposition trials were performed on an unknown SiC material remaining from an unrelated program. Early results were encouraging, with good film adhesion, but only a weak ultrasonic signal. Low electrical conductivity of the SiC was suspected, and an investigation of higher-conductivity SiC was begun. Additional films were deposited on three additional SiC materials: Hexoloy SG, a sintered material produced by Saint-Gobain Ceramics; Pure SiC-LR, a CVD material from Coorstek Ceramics; and Performance SiC, a CVD material from Morgan Advanced Ceramics. Figures 3-6 show films successfully deposited on the various SiC substrates. No special surface conditioning or preparation was required to achieve adhesion on these materials. At least two films were attempted on each material; films deposited on Pure SiC- LR, had the best adhesion, but all materials showed reasonable adhesion. Additional depositions on WC were also performed in the second year of the program.

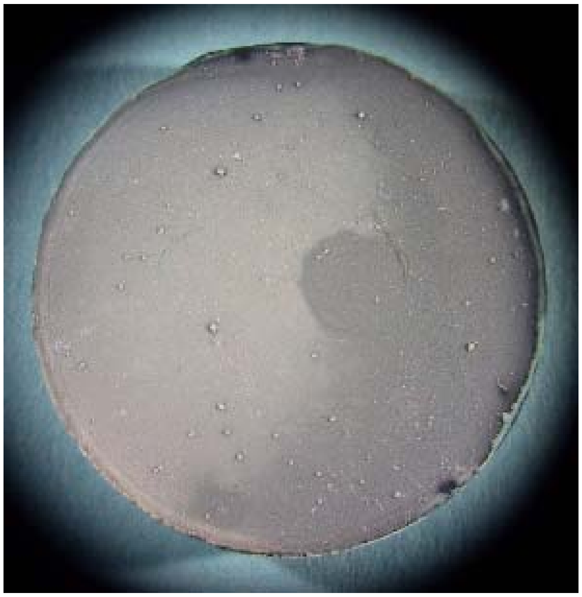

Figure 3. As-deposited AlN film on SiC.

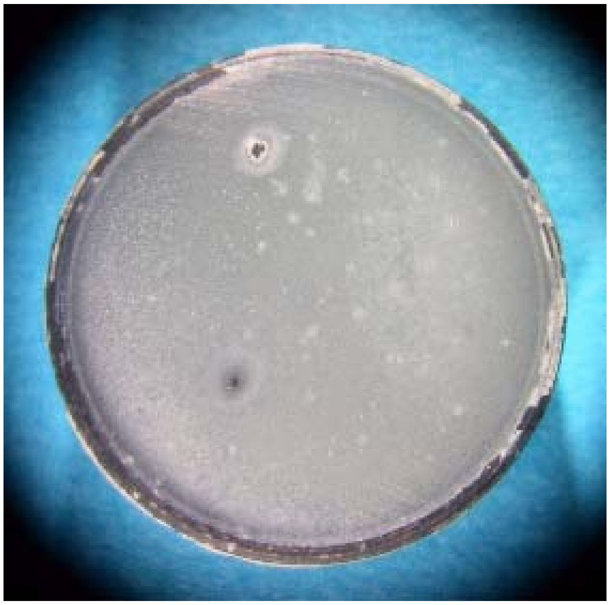

Figure 4. Rough-polished AlN film on *Hexoloy SG*.

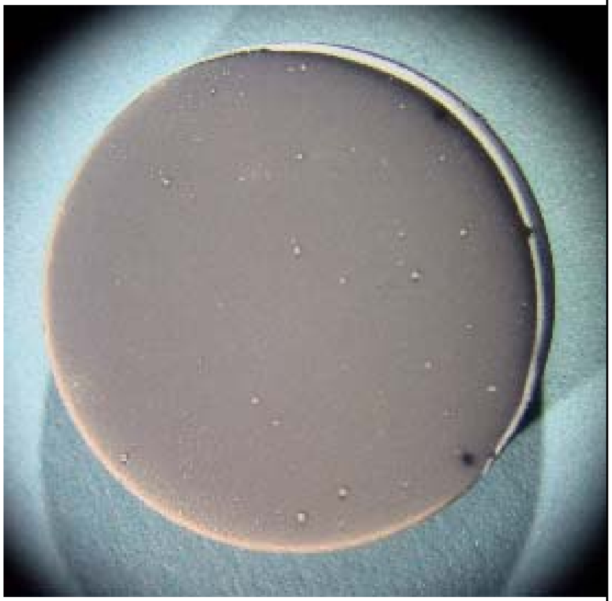

Figure 5. As-deposited AlN film on *Pure SiC-LR*.

Figure 6. As-deposited AlN film on *Performance SiC*.

Later experiments, discussed below, showed that the adhesion is not maintained when the substrate/film is thermally cycled. As a result, two additional substrate materials were investigated. The first was a cobalt-coated SiC, with the intent being to replicate some of the surface chemistry of the tungsten carbide. This coating was unsuccessful; the AlN spalled from the surface immediately, as shown in Figure 7. A second material was an electrically conductive grade of AlN procured from NGK Insulators. Deposition on this material was successful and the film is show in Figure 8. The AlN substrate was not further pursued because, like WC, it is not more oxidation-resistant than the piezoelectric AlN films.

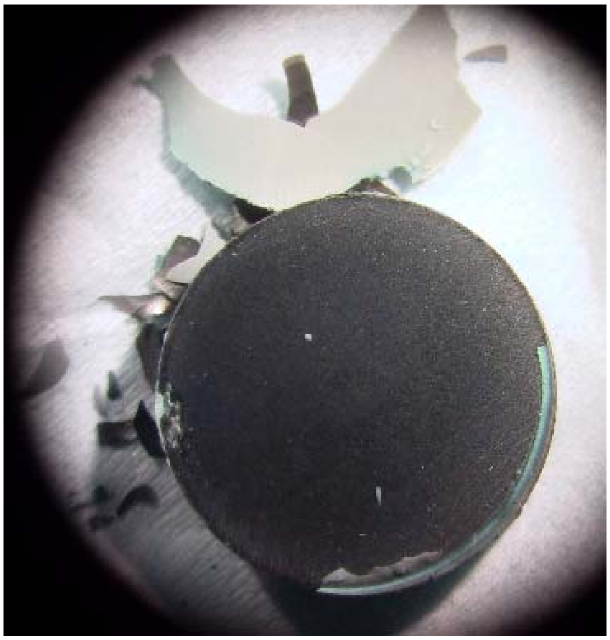

Figure 7. AlN film spalled from cobalt-coated SiC

Figure 8. As-deposited AlN film on conductive bulk AlN substrate.

Piezoelectric AlN Film Evaluation

Physical evaluation, primarily film adhesion to the substrate, is the initial evaluation criteria. The thin (<0.1 mm) AlN films require the substrate for deposition and subsequent handling; the conductive substrates also function as one of the electrodes used to energize the AlN films. Films on WC show good adhesion, maintaining adhesion when polished and when thermally cycled to 800°C or above. Some chipping at the edge is commonly the only delamination seen. At the other extreme, the films deposited on titanium showed poor adhesion upon heating, often spalling completely from the substrate and causing titanium to be removed from consideration as a candidate substrate material.

The AlN films on SiC showed mixed results. Most films exhibited good initial adhesion, as shown in Figures 3-6. Some of these were rather weakly bonded, however, and underwent significant damage or delamination upon polishing. Figure 9 shows a typical example with edge damage and a delamination (white area) extending from under a pore. Figure 10 shows more severe damage incurred on a different SiC material. The Hexoloy SG and Pure SiC-LR materials both showed acceptable adhesion and were further evaluated for ultrasonic properties and thermal cycling resistance.

Figure 9. Minor localized polishing damage on Hexoloy SG substrate.

Figure 10. Polish-damaged AlN film on Performance SiC substrate.

Both materials showed poor adhesion upon thermal exposure. Delaminations usually began at an edge or existing defect, and spread quickly under the film, which split and buckled. Figure 11 shows the same film as Figure 9, in its as-received, polished, and then after heating to only 400°C. The pore in Figure 9 is at the top of Figure 11, just left of center. The darker areas are still well adhered, but more than half of the film has delaminated and cracks are visible in the center of these regions. An AlN film on Pure SiC-LR, which had only minor edge delamination prior to heating, experienced even more severe damage. Figure 12 shows this film before and after heating.

Figure 11. Delaminated AlN film on Hexoloy SG substrate after heating.

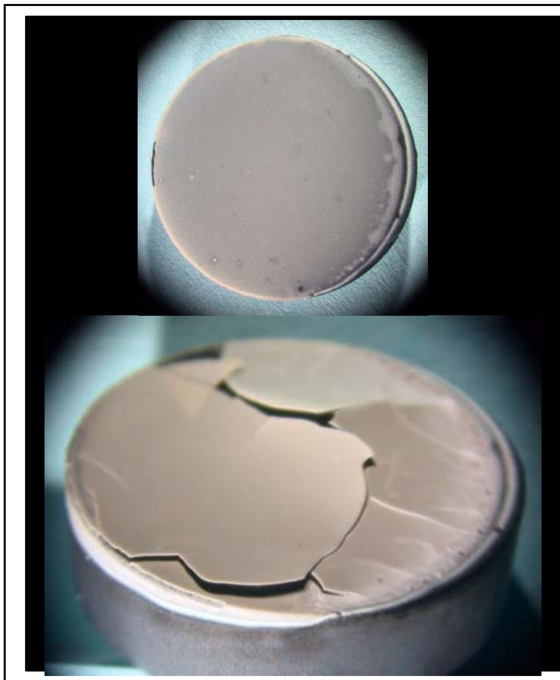

Figure 12. Massively delaminated AlN film on Pure SiC-LR substrate before and after heating.

Some of the failed films were subjected to EDS analysis to try to determine the failure mode after delamination. Early AlN films on WC that delaminated had been found to actually pull some WC material off the surface of the substrate, indicating a strong bond. AlN films delaminated from titanium metal were found to have two distinctive layers, with an interface layer that remained attached in some cases, and a bulk AlN layer which spalled. This was not the case for the various films on SiC- the AlN separated cleanly from the SiC.

Testing for ultrasonic properties was performed on “good” films, after polishing and before heating. Figure 13 shows the Utex 340 ultrasonic square wave pulser/receiver instrument was used with an Agilent 54622A digital oscilloscope for these tests. The Utex 340 incorporates programmable low pass and high pass filtering, and can supply pulses of 5-80 ns with an amplitude of 100 to 500 V. Test voltages near 300 V were used for most films unless electrical shorting near damaged areas forced the use of lower voltages. For most testing, the film was placed on water-based ultrasonic couplant on a flat metal electrode. A two-pronged probe from the pulser/receiver was then touched to this stack, with the ground on the metal electrode and the signal lead on the film’s substrate. Pulse width, gain, and receiver filters were adjusted to maximize the signal on a film-to-film basis.

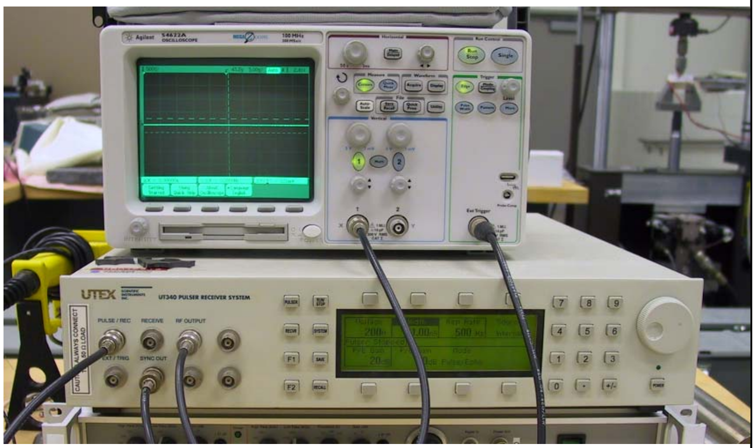

Figure 13. Utex 340 Pulser-Receiver and Agilent Oscilloscope.

The frequency content of the first clear echo from the back of the substrate was used to determine the natural frequency of the film. The echo from the far surface of a 25.4 mm thick, 17-4PH stainless steel cylinder with parallel faces, used as the metal ground electrode, was also used to provide a film-to-film strength comparison without effects from the substrate geometry. Pulse width and gain settings were adjusted as needed. Averaging of multiple pulses was used to increase the signal to noise ratio. Typical ultrasonic waveforms were shown in Figures 1 and 2.

Initial results with AlN films on SiC were disappointing but showed promise. The films were producing ultrasonic energy, but weakly. Further polishing of both the AlN films and the back surface of the SiC substrates did not improve the signal strength. Time-averaging was used to improve the S/N ratio of the ultrasonic signal. A breakthrough was made in signal strength when it was discovered that the marginal electrical conductivity of the SiC, combined with an oxide layer in some cases, was leading to an unacceptably high lead resistance between the substrate and the ultrasonic pulser-receiver. This was overcome by placing a metal disk against the back of the substrates to act as a wide electrode. Signal strength improved by ~20 dB, producing ultrasonic signals similar in amplitude to those form AlN on WC, but with substantially better bandwidth. The results section below contains details of the ultrasonic evaluation.

High Temperature Ultrasonic Coupling

Based on information gathered in the literature survey during the first year of the program, high temperature coupling experiments were designed to evaluate the use of molten glass and metal foil to couple ultrasonic energy between a transducer and a test object. Successful high temperature ultrasonic coupling is required for overall program success; commercial ultrasonic couplants are not marketed for use at these temperatures because ultrasonic transducers for very high temperatures are also not currently marketed.

The most basic coupling experiment was to test the molten glass coupling. Two electric resistance band heaters encircling a 25 mm diameter test cylinder 100 mm long provided heat. A K-type thermocouple welded to the cylinder near the top measured temperature. Powdered glass was placed on the cylinder, followed by an AlN film on a WC substrate. A second thermocouple welded to the substrate provided a second temperature measurement, and the interface temperature was estimated to be the average of these two thermocouples. Another stainless steel cylinder, this one only 25mm long, was placed on top to provide a small compressive load to the interface. This entire test stack was generally wrapped with an insulating blanket to accelerate the heating time and to maintain a more uniform temperature.

To produce and receive ultrasound, the thermocouple wires were disconnected from the temperature readout and connected instead to the Utex pulser/receiver, with the large cylinder acting as the ground and the substrate thermocouple acting as the signal lead. Upon heating past its melting point, the powdered glass liquefied and did function as an ultrasonic couplant. While the AlN showed some signs of chemically reacting with the glass, it was not physically damaged by the resolidification of the glass.

In order to test metal foil coupling, the same apparatus and procedure were followed, except that a larger compressive load was required; a servohydraulic load frame with water-cooled compression platens applied this load as shown in Figure 14. Ceramic and mica sheets placed between the platens and the test stack described above prevented the rapid conduction of heat into the cooled platens. A spherical washer at the top platen accounted for any misalignment in the test stack. While the load frame used in the experiments was capable of applying 500 kN, the lowest range was selected and the largest force used in this testing was 12 kN. After the load was set, the servohydraulic controller maintained the load, accounting automatically for displacements caused by thermal expansion when the test stack was heated. Aluminum, silver, and gold foils were tested for coupling.

Figure 14. Coupling test stack in load frame.

SECTION 4
RESULTS AND DISCUSSION

Piezoelectric AlN Film Deposition

Task Description: The deposition of AIN films onto high temperature substrates will be studied and improvement made with respect to process and yield. Disposition of films will initially be done by an elevated temperature chemical vapor deposition process. An alternative deposition process, pulsed laser deposition (PLD), will also be attempted. Temperature of the substrate and PLD pressure will be varied to create an AIN film with piezoelectric behavior.

Subtask 1A: Conduct deposition of thick AIN film by chemical vapor deposition. Nov 2002-May 2005. The CVD process continues to produce piezoelectric AlN films. Additional films were created in the second year of the program on tungsten carbide, silicon carbide, and conductive aluminum nitride as described in the "Experimental" section above. Additional depositions in the final year of the program will create AlN films for demonstration sensors and for a continued investigation into improving adhesion on SiC substrates.

Subtask 1B: Identify the promising high temperature substrates with regard to thermal expansion, cost, machinability, and the ability to shape the substrate. Nov 2002-May 2004. Investigation into alternative substrate materials proved challenging, as noted in the first year's report. In the second year of the program, several grades of SiC were investigated with reasonable results. The electrical conductivity issues with SiC have been overcome, but the AlN films are still not durable under thermal cycling. Deposition also was successful on a novel, electrically conductive grade of sintered aluminum nitride. Titanium silicocarbide looked promising from a machinability standpoint, but it's relatively high coefficient of thermal expansion is too high to be compatible with the AlN films. Porous tungsten carbide looked promising, as the porosity would improve the acoustic impedance match with AlN while attenuating substrate echoes, but was not obtainable commercially. The AlN did not adhere to a cobalt-coated SiC substrate.

In the final year of the program, deposition will be attempted on several additional substrates. A different division of Saint-Gobain from the one that produces the Hexoloy SiC material has agreed to team with UDRI to further investigate coating SiC with AlN. They will be providing siliconized SiC and porous SiC substrates, as well as analysis of the interface chemistry and suggestions for surface treatments. In addition, deposition will be attempted on WC and SiC coated with WC using a flame-spray process. This ought to attenuate substrate echoes, while possibly acting as an interface to adhere the AlN and SiC.

Subtask 1C: Experimentally explore the feasibility of AIN deposition onto identified new substrate materials such as graphite, alumina graphite, silicon carbide, titanium diboride, and molybdenum disilicide. January 2003-May 2005. Deposition has been achieved to date on commercially pure (Grade 2) titanium, tungsten carbide, various grades of silicon carbide, and a bulk aluminum nitride. For elevated temperature use, only the WC appears practical, but it is likely that further experimentation with SiC substrates will allow its use as well. Final substrate selection for the demonstration sensors will be delayed as long as practicable. WC can be used beyond 1000°C if the sensor is constructed to exclude oxygen; the AlN films will likely also benefit at temperatures above 800°C.

Subtask 1D: Investigate the feasibility of using pulsed laser deposition of the AIN films onto substrates. The substrate pressure and temperature will be varied to determine conditions conducive to producing films specific for this high temperature sensor application. January 2003-January 2004. This subtask was completed in the first year of the program. AlN film deposition using PLD was demonstrated. The process is not practical for full-scale sensors at this time, but might be useful in the future for the repair of CVD films or for miniature sensors.

Piezoelectric AlN Film Evaluation

Task Description: An evaluation of films deposited in task 1 will be completed in task 2. The films and high temperature substrates will be characterized structurally and ultrasonically. Characterization of the films will include the adhesion, oxidation, and continuity of the films when exposed to both room temperature and high temperature atmosphere. The ultrasonic properties of the films will also be determined by investigating the frequency, bandwidth, and signal strength at elevated temperatures. Using a scanning electron microscope with X-ray diffraction the surface and subsurface of the films will be analyzed to provide data on the crystal structure, defects, and bonding.

Subtask 2A: Characterize films by examining the thermal and physical properties of the films. Structural evaluation includes adhesion, oxidation, and continuity of the films as a function of time, temperatures up to 1000°C, and exposure to physical damage. December 2002- June 2005. Characterization to date has included baseline films on tungsten carbide and films on two new substrates, titanium and silicon carbide.

The films on titanium were found to be inferior upon heating above 100°C and not durable enough for deployment in a sensor. Heating causes films on titanium to disband from the surface of the substrate, rendering them useless as transducers. Surface roughening and additional cleaning to promote adhesion was ineffective. Most likely, the mismatch in thermal expansion between the titanium and AlN causes large thermal stresses at the interface, breaking the bonds formed during deposition. While early disbonded films on WC generally pulled away a small amount of the substrate material when they failed, the opposite effect was observed on the titanium: there was often a thin coating remaining on the titanium when the film disbonded. The poor results of titanium substrates pointed away from using other materials with a large expansion mismatch with the AlN, and toward lower expansion substrate materials.

The requirement for a low thermal expansion material points to ceramics, but most ceramics are electrical insulators; a conductive substrate is required to act as an electrode on one side of the films. Some forms of silicon carbide have adequate conductivity from room temperature up, and this material was tried next. The acoustic impedance of SiC is close to that of AlN, allowing an AlN film on a SiC substrate to produce a high bandwidth ultrasonic transducer with very little ringing of the ultrasonic pulse, as was shown in Figure 2. As discussed in the *Experimental* section, film adhesion to SiC was good initially, but thermal damage accumulated easily, in some cases as badly as with titanium substrates. The best results were with the sintered material; the

AlN seemed to form a weaker bond with the CVD material, even though it should have been more pure. This points towards possible mechanical bonding with the very different microstructure of the sintered material, which will be investigated further in the final year of the program, along with the evaluation of additional SiC materials.

Subtask 2B: Characterize films by evaluating the ultrasonic properties of the films and substrates. Ultrasonic evaluation includes frequency, bandwidth, and signal strength at ambient and elevated temperatures. December 2002- June 2005. The new AlN films on SiC possess ultrasonic properties similar to previous films on tungsten carbide and titanium. Films thick enough to have center frequencies below 30 MHz and substantial energy below 10 MHz continue to be produced. The acoustic impedance of SiC is even a better match for AlN than that of titanium, and the resulting films have a very desirable wide bandwidth with minimal ringing. Ultrasonic data for the films on SiC has been collected only at ambient temperature due to the elevated temperature adhesion issues with films produced to date. The early problems with making adequate electrical connections to the SiC substrates have been overcome to facilitate this testing.

Figures 15 and 16 show strong signals from an AlN film on SiC. The signal in Figure 15 is the second reflection of the sound from the back surface of the SiC substrate. Substrate echoes are only moderately useful to evaluate signal strength, but may be used to evaluate bandwidth and center frequency. This film has a -6dB bandwidth of 75% and a center frequency of 32 MHz, as shown in Figure 16. Figure 17 shows a reflection of a pulse from the same film off an external, 25mm thick block of 17-4 PH stainless steel. This block has polished, parallel faces, so the amplitude of the echo in it may be used to compare the strength of films to one another. The signal in Figure 16 is ~350mV p-p, 15 MHz center frequency, and was recorded with only 20dB of gain with a 200V excitation pulse. These waveforms are representative of the signals produced by the stronger ALN films on SiC. The reason for the change in center frequency is that the test block material acts like a low pass filter, attenuating the higher frequency components of the signal, which is also attested to by the reduced amplitude in Figure 16. The high bandwidth of the films on SiC spreads the ultrasonic energy to lower and higher frequencies, producing more energy likely to get through attenuative real-world materials.

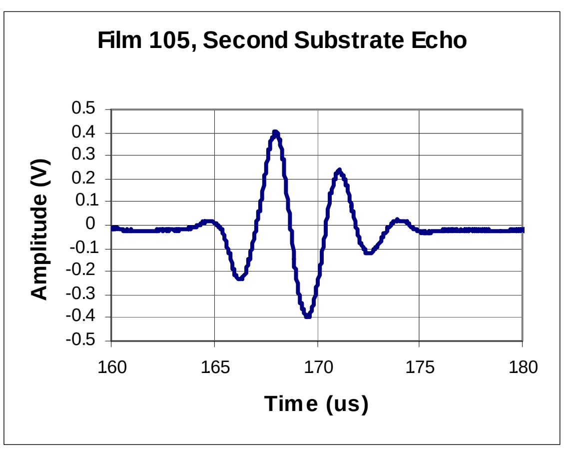

Figure 15. Wideband ultrasonic signal produced by AlN film on SiC substrate.

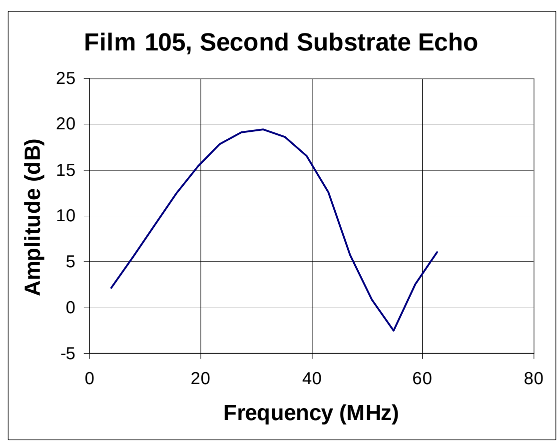

Figure 16. Frequency spectrum of ultrasonic signal in Figure 15.

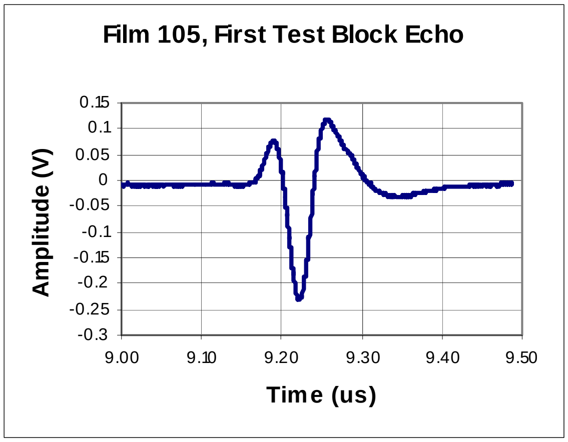

Figure 17. The same signal, reflected through a 25mm test block.

Subtask 2C: Characterize films using x-ray diffraction and scanning electron microscopy to examine the piezoelectric crystal structure, defects, bonding difficulties, and other material issues. March 2002-December 2004. Much of this evaluation was postponed to be used on AlN films intended for eventual incorporation into a sensor; those that have been failing at temperature do not meet this criterion. However, electron dispersion spectroscopy (EDS) techniques have proven useful along with electron microscopy to characterize the nature of the film delaminations caused by thermal cycling. As noted above, indications are that a clean separation occurs between the AlN and the SiC in most failures, indicating a bond that is primarily mechanical in nature.

Subtask 2D: Relate substrate surface preparation to film quality (adhesion, defective areas, film thickness uniformity, and orientation of the piezoelectric crystals). December 2002-December 2004. Various surface preparation methods were used on titanium substrates, eventually causing adhesion at room temperature, but not robust enough to overcome the thermal expansion mismatch. Minimal surface preparation of silicon carbide substrates has been required, with good films resulting from no preparation or from polishing and cleaning the as-received substrate material. Oxidation of the SiC may be an issue, and the presence of an oxide layer at the surface prior to deposition of the AlN will be evaluated. No relationship has been discovered to date between surface condition and "good" and "bad" areas of a film. Surface preparation also does not appear to have a significant effect on crystal orientation in the films.

High Temperature Coupling

Task Description: A literature survey will be made of methods to conduct ultrasonic coupling at elevated temperatures and those that are most suited to equipment health monitoring. Those methods that appear most suited for the application will be verified in the laboratory, in particular, the effect of the coupling layer on the impedance matching will be examined.

Subtask 3A Perform literature survey re high temperature coupling. January 2003-August 2003. This subtask is complete. The literature survey was completed by UDRI with the help of a mechanical engineering student employed as part of our cooperative education program, as summarized in the first year's report. Metal foils and molten glasses were selected as the methods with the highest probability of success and were further evaluated experimentally.

Subtask 3B Design high temperature apparatus for testing films and coupling to test objects. August 2003-December 2003. Based on the literature survey, the technical community appeared to have had limited success in developing ultrasonic coupling methods suitable for use at high temperatures. Accordingly, this subtask was expanded through the second year of the program, and formed the basis of an undergraduate Honors Thesis for the student who performed the literature survey. A simple test fixture was designed and constructed as described in the *Experimental* section and shown in Figure 14. Tests were performed on metal foil coupling, beginning with aluminum and progressing to silver and gold, with gold chosen for future high temperature work due to its oxidation resistance and melting point above 1000°C.

Figures 18-21 compare the first echo in the test block using a commercial water-based couplant at room temperature to the echoes received using the various foil couplants at elevated temperatures. Note that good coupling is achieved, even at temperatures approaching the

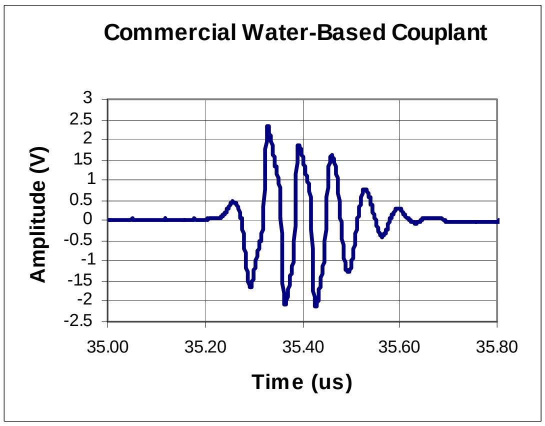

Figure 18. First reflection in 100 mm test block, water-based couplant, room temperature.

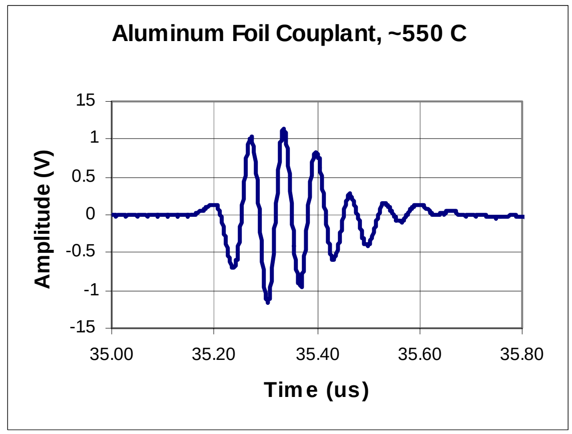

Figure 19. First reflection in 100 mm test block, aluminum couplant.

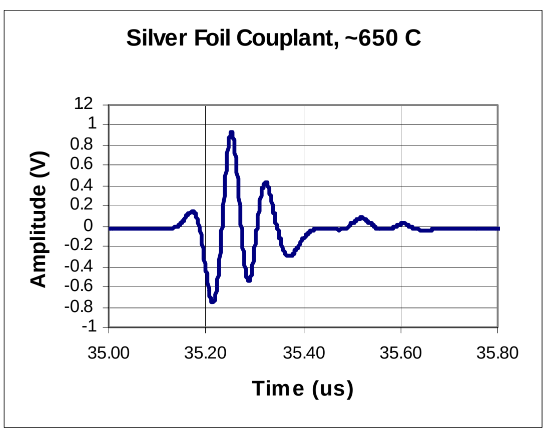

Figure 20. First reflection in 100 mm test block, silver couplant.

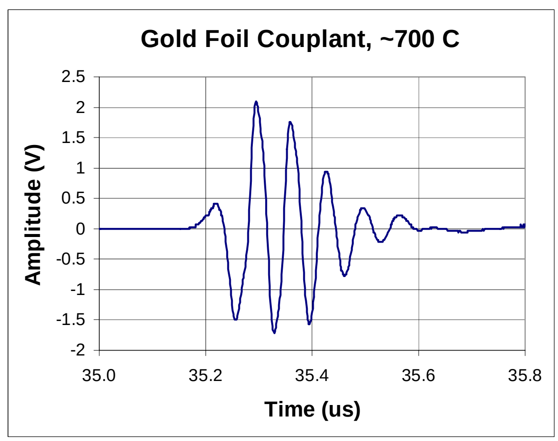

Figure 21. First reflection in 100 mm test block, gold foil couplant.

melting point of the aluminum foil. Aluminum and silver foils showed some degradation with time, likely the result of oxidation. The strongest signals were achieved using the gold foil In general, some degree of coupling occurred as soon as a compressive load was applied to the test fixture. Upon heating, the softening of the foils improved the coupling, and a high degree of coupling was maintained even after cooling and the removal of almost all the compressive load. A single AlN film on a tungsten carbide substrate was used for all the foil couplant tests.

The aluminum foil couplant was used to evaluate the effects of compressive load on coupling effectiveness for foil couplants. Figure 22 shows an increase in coupling effectiveness with load up to about 15 kN, corresponding to a pressure of about 10 MPa. Note that this value cannot be compared to the yield strength of the aluminum because creep effects dominate at these temperatures and the thin aluminum layer is in a triaxial state of stress, corresponding more closely to a hydrostatic pressure than uniaxial compression. This shows that for foil coupling, there is an ideal load that should be reached, beyond which additional loading is unnecessary. As mentioned earlier, this load need not be maintained after initial loading and heating.

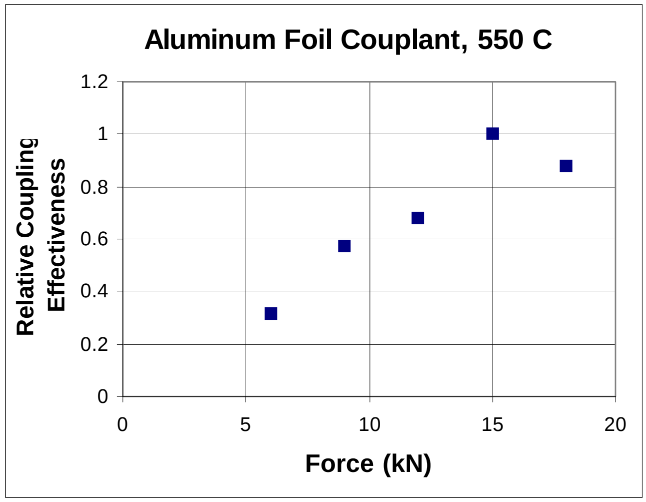

Figure 22. Coupling effectiveness as a function of compressive load.

Aluminum foil couplant was also used to evaluate the effects of temperature on coupling effectiveness for foil couplants. The summary data in Figure 23 show a weak dependence on temperature compared to the dependence on compressive load. Good coupling was achieved at 12 kN essentially independent of changes in temperature. At the lower load levels, increasing the test temperature to between 500°C and 550°C improved the coupling, although further increases may be detrimental. The best route for coupling a transducer appears to be applying adequate load and heating to within a specified softening range. Additional couplant work in the third year of the program will involve determining these parameters for gold couplant. The use of "gold leaf," thinner than the foils used to date and commonly used for artistic purposes, will also be investigated. The thinner gold leaf has the potential to reduce the ultrasonic effects of the couplant on the signal while simultaneously reducing the cost of the couplant.

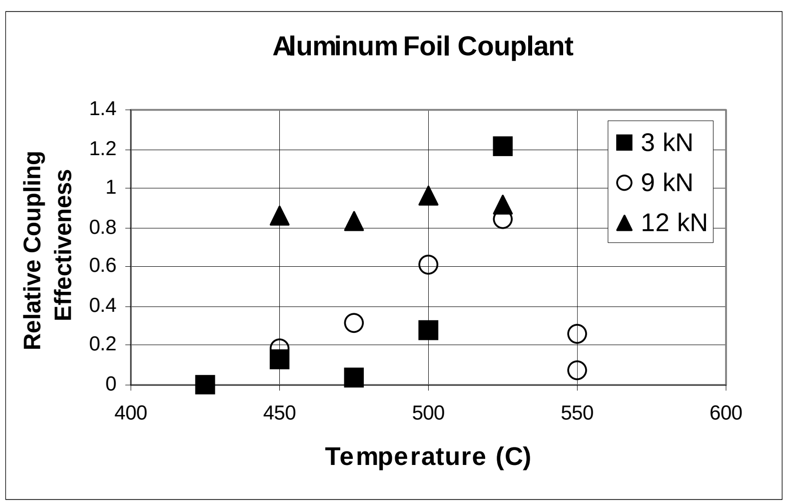

Figure 23. Coupling effectiveness as a function of compressive load and test temperature.

Testing was also performed using molten glass couplant. Figure 24 shows the glass powder (white) in the test fixture prior to heating; Figure 25 shows it on the AlN film after the test was completed.

Figure 24. Glass couplant test setup prior to heating.

Figure 25. Glass couplant after test.

The AlN film was separated from the test block while the glass was still soft. The ultrasonic coupling within the appropriate temperature range was adequate, as shown in Figure 26, although this data was acquired at 10 dB greater gain and still has a lower amplitude than the earlier plots. The molten glass was found to be effective in only a narrow temperature range for the two glass compositions tried. Below this temperature, the glass layer did not soften and thin, preventing ultrasonic coupling and reducing the electrical field strength driving the piezoelectric film. At much higher temperatures, the glass began to react chemically with the air and with the steel test block. Figure 27 shows the same setup, 30 minutes later, with the temperature slowly increased. The signal strength is greatly diminished. Molten glass appears to be an effective couplant for limited use, but the metal foil method does not suffer from material incompatibility issues, has a wide operating range, and has been chosen for use for the remainder of this program.

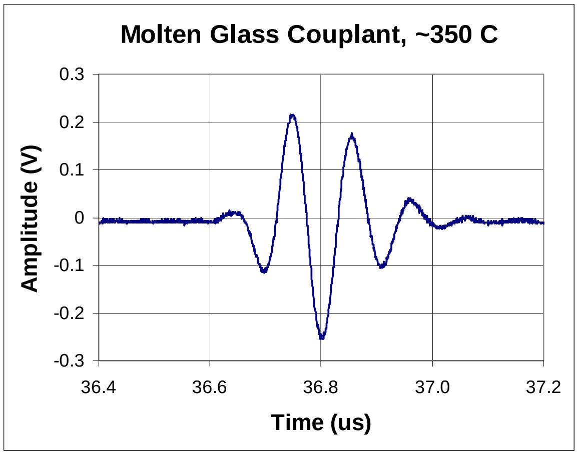

Figure 26. Glass-coupled ultrasonic signal.

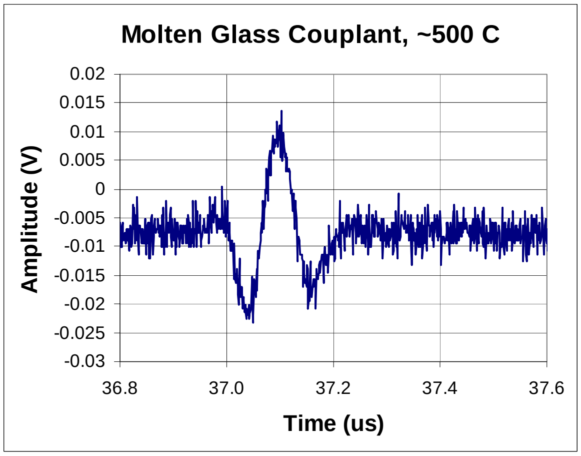

Figure 27. Glass-coupled ultrasonic signal.

Equipment Health Monitoring Demonstration

Task Description: A specific application of an ultrasonic piezoelectric sensor for monitoring equipment health will be selected. Requirements for the specific application will be defined and used to direct the design and fabrication of the sensor. The sensor design will focus on the AlN film, electroding, ultrasonic coupling, high temperature wiring. The feasibility of the sensor will be demonstrated in the laboratory under conditions that simulate the specific application.

Subtask 4A Identify specific application to test AlN film based ultrasonic sensor. Application must be able to be mimicked in the laboratory to demonstrated feasibility of sensor. May 2003- May 2004. Three candidate applications have been identified. The first is as a point sensor to monitor the thinning of the inner layer of refractory in a coal gasifier. If several such sensors were located in likely areas of degradation, this system could be used as a continuous monitor of refractory wear during service. However, there is a strong possibility that the high frequency ultrasound from the sensor cannot be used in the ceramic due to its microstructure. Efforts are underway to procure a sample of a typical gasifier refractory to evaluate it ultrasonic attenuation.

A second candidate application is to monitor the condition of a thermal barrier coating applied to a metal part. Signal conditioning for this application would be simple, consisting initially of a go-no go test to determine if the coating remains bonded to the base material. Unlike the signal conditioning, the sensor construction would be difficult. Most components using thermal barrier coatings are relatively small, sensitive to the addition of a sensor mass/volume, and in many cases are rotating. This is most likely not the best candidate for an initial test.

The third candidate application, and the most promising, is a monitor of process equipment such as a heat exchanger. Ultrasonic inspection is commonly performed on such equipment during plant shutdowns. Welds and corrosion-sensitive areas are often trouble sources. Clamping one or more transducers to a pipe heated to the test temperature can simulate these conditions rather easily. Transducers will likely be developed to look for wall thinning or cracking.

Subtask 4B Set up laboratory for feasibility demonstration. May 2004- December 2004. Because of the earlier uncertainty of the transducer makeup, regarding substrate and coupling, the start of this subtask was delayed into 2005. It will proceed in parallel with Subtasks 4C and 4D in the final 9 months of the project.

Subtask 4C Design and fabricate sensor specific to the application including the AlN film, electroding, ultrasonic coupling, and high temperature wiring. May 2004-June 2005. Transducer design work began in the second year of this project. Metal-sheathed thermocouples and coaxial cable have been ordered and received to evaluate for transmitting the ultrasonic signals from the hot transducer back to signal-conditioning electronics. A standard laboratory pulser-receiver will be used for the majority of testing, but a prototype circuit is under development to demonstrate a low-cost alternative for an installed sensor. The transducer design is set up to include metal-foil coupling, either directly to a test object, or coupled into a short delay line and then into the test object. An external means of applying pressure will be required, as will a housing that can withstand the test environment. This general design will be quickly adapted to the specific test application once its conditions are established.

Subtask 4D Evaluate performance of sensor system at elevated temperatures (700-1000^{o}C) in selected application. December 2004- August 2005. This subtask has not yet begun.

SECTION 5
CONCLUSION

The project is on schedule to complete the desired tasks within the program period and budget. Through the second year, expenditures have been approximately 50% of the project total, but work in the final year, especially the demonstration project, will increase the rate of expenditures to match the overall budget. The student who has performed much of the couplant work will be graduating and another student will be employed full-time in the coming summer on the project. At this point, the high-temperature coupling issue is under control with the use of gold foil. The final year will focus on continued sensor improvement and the implementation of a sensor demonstration in the laboratory.

To perform the sensor demonstration, the major task remaining is to determine a final substrate material and configuration. Silicon carbide will be used if the adhesion issue can be overcome; otherwise tungsten carbide will be retained and the sensor will be sealed to exclude air and prevent oxidation. Substrates will be shaped or otherwise modified to reduce or eliminate internal echoes. Depositions of AlN films will continue in support of these efforts.

Other activities during the year will include refinement of the metal foil coupling method and the sensor demonstration itself. Several transducers will be built for the demonstration, designed to be robust enough to deploy in industry with minimal changes. A specialized prototype circuit to drive the transducers without expensive general-purpose instrumentation will also be investigated.

Research to date has not been complete enough to be submitted to conference proceedings or publication, but plans to do so include: (1) A student thesis and associated presentation regarding high temperature ultrasonic coupling. (2) Conference papers on the high temperature ultrasonic transducer advancements during the program and the elevated temperature metal foil coupling are planned for the Annual Review of Progress in Quantitative Nondestructive Evaluation (QNDE) conference in August 2005. (3) A conference paper or journal article is also planned regarding the application of the final sensor design to the selected demonstration application.