

THE REALIZATION AND TESTING OF A SAPPHIRE PRESSURE SENSOR MANUFACTURED BY LASER MICROMACHINING AND THERMOCOMPRESSION BONDING

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ABSTRACT

This paper describes the fabrication process, packaging, and testing of a sapphire optical pressure sensor developed for measurements in hypersonic flows. Ultrashort-pulse laser micromachining was utilized to etch a 5-mm-diameter cavity feature into the sapphire supporting structure, which was attached to a 30- μm -thick substrate via thermocompression bonding to form the sapphire sensing element. Measured sensor performance metrics at ambient temperature included a sensitivity of 0.44 $\mu\text{V}/\text{Pa}$ and a bandwidth of at least 6.7 kHz. The sensor was calibrated up to a temperature of 880°C by utilizing a plane-wave tube housed in a tube furnace.

KEYWORDS

Sapphire, Pressure Sensor, Optical, High-Temperature, Laser Micromachining.

INTRODUCTION

Measurements of pressure fluctuations at $>1000^\circ\text{C}$ are needed to characterize the boundary layer of hypersonic flows [1]. Specifically, measurements of trapped acoustic waves (i.e., the second-mode instability) within the boundary layer of a hypersonic flow can provide insight into the transition from laminar to turbulent flow [2]. Based on wind-tunnel experimentation of cone-shaped models, a fully turbulent boundary layer can increase the heating rate by up to a factor of eight [3]. Pressure measurements of instability waves within hypersonic boundary layers can provide insight into the efficient design of a hypersonic vehicle's heat shield to minimize excess take-off weight.

Second-mode instability waves for hypersonic flow over a cone at zero angle of attack have a predicted frequency of 325 kHz [4], necessitating the miniaturization of sensing equipment to spatially resolve these physical structures with high accuracy. Although pressure sensors have been developed with sufficient bandwidth to capture these and other high-frequency fluctuations [5], the thermal capability of commercially available solutions significantly lags the target continuous operating temperature for real-world hypersonic flows. Probe tubes have been utilized to recess sensors from the harsh environmental conditions; however, the tube can attenuate the pressure wave due to visco-thermal dissipation near the walls of the tube [6]. Water-cooling is another thermal mitigation strategy used to bring the local temperature of the sensor to within standard operating conditions. This adds packaging complexity and can introduce thermal gradients to the sensor which makes it challenging to accurately correct the sensor's response [6].

To overcome these limitations, a fiber-optic lever transduction scheme was implemented to enable in situ measurements without the need for water-cooling. Sapphire was used as the material platform due to its high thermal robustness and its commercial availability as both a bulk substrate and an optical fiber. A prototype device, developed by Griffin et al. [7], demonstrated the working principles of a sapphire optical pressure sensor utilizing this transduction scheme, but no thermal testing was performed. Following this development, advanced manufacturing methods and

a high-temperature calibration process were implemented. The major limitations of these continued efforts were thermal limitations of 500°C due to diaphragm delamination [8] and diaphragm buckling due to residual stress produced by the bonding process [9]. This paper details the realization and testing of a sapphire optical pressure sensor that was calibrated up to 880°C.

DESIGN AND MODELING

Sensor Design

The sapphire sensing element is shown in Figure 1. It consists of a sapphire diaphragm suspended above the cavity of a sapphire supporting structure, with the two substrates joined via a titanium/platinum bonding layer. A sapphire fiber is coupled to the bottom of the sensing element with a zirconia ferrule, which is used to precisely control the distance between the fiber and diaphragm. A titanium/platinum film stack on the backside of the diaphragm enables reflection of the optical signal transmitted by the fiber. As the diaphragm deflects, the optical path length between the fiber and diaphragm changes and this modulates the intensity of the recaptured optical signal. A single fiber is used for this fiber-optic lever transduction scheme to simplify packaging at the expense of a reduced sensitivity over bundled fiber arrangements.

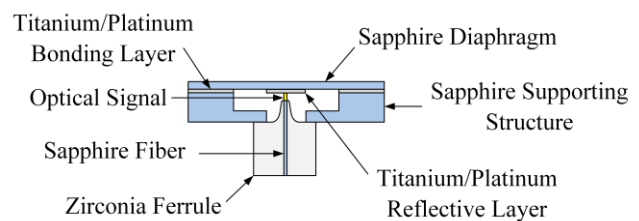


Figure 1: Schematic of the sapphire sensing element.

Frequency Response Modeling

Lumped-element modeling was utilized to predict the frequency response of the pressure sensor at ambient temperature and at 880°C. Details of the theoretical derivation of the model are provided by Vera et al. [10]. Figure 2 shows the magnitude of the predicted frequency response ($|H|$) of the sapphire sensor. The predicted ambient sensitivity and resonant frequency are 0.31 $\mu\text{V}/\text{Pa}$ and 24.2 kHz, respectively. At 880°C, thermal expansion causes an increase in diaphragm thickness and diameter as well as an offset in fiber-diaphragm separation. Additionally, the speed of sound and dynamic viscosity of air increase, and sapphire's elastic modulus decreases. These factors contribute to an overall 1 dB increase in sensitivity at 880°C, accompanied by a shift of the resonant frequency to 22.9 kHz.

FABRICATION AND PACKAGING

Subtractive Manufacturing

A fabrication process flow of the sapphire sensing element is presented in Figure 3. The structure shown in Figure 3(a) is the 10 mm x 10 mm x 0.5 mm R-plane sapphire supporting structure that was cut to size from a 100-mm-diameter wafer by a diamond dicing

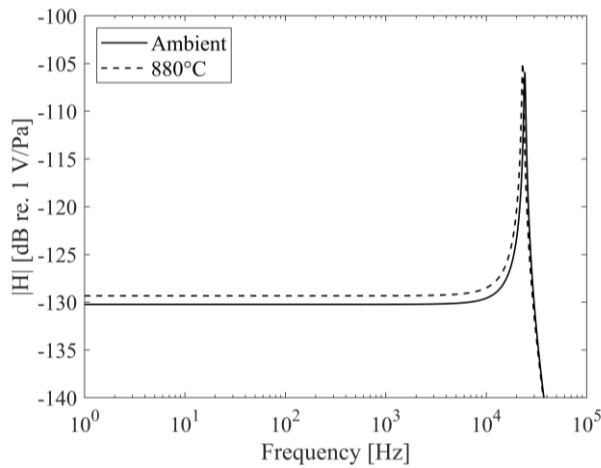


Figure 2: Predicted frequency response of the sapphire optical pressure sensor at ambient temperature and 880°C.

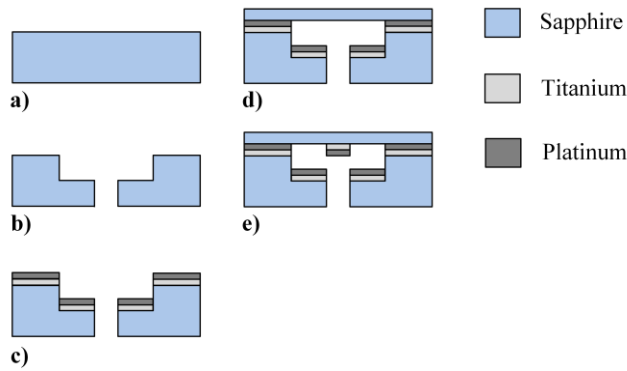


Figure 3: Sapphire sensing element fabrication process flow.

saw blade. Dry and wet chemical fabrication processes are not effective for etching features into sapphire since it is chemically inert [11]. Alternatively, ultrashort-pulse laser micromachining has been demonstrated as a viable method for etching precise features into sapphire substrates [8]. In this pulse regime, the effects of thermal conduction are significantly diminished which reduces the heat-affected zone within the workpiece [12].

An Oxford J-355PS laser micromachining station, with a pulse duration of 10-15 ps, was used to etch features into the sapphire supporting structure. A 5 mm diameter cavity was etched to a depth of 350 μm in the center of the sapphire supporting structure. Following this, a 1.5 mm central through-hole was machined for integration with the 2.5-mm-diameter zirconia ferrule. These two etching steps are shown in Figure 3(b). A 422- μm -tall step feature was laser-micromachined onto the ferrule to position the end face of the sapphire fiber 78 μm away from the diaphragm. At this position, the static mechano-optical sensitivity is at its maximum. This 78 μm distance was quantified by an experiment characterizing the mechano-optical sensitivity of a sapphire fiber that was transversely displaced from a mirror, as detailed by Vera et al. [10]. Images of the laser-micromachined sapphire supporting structure and zirconia ferrule are shown in Figure 4(a) and Figure 4(b), respectively.

Additive Manufacturing

A metallic film stack was deposited on the topside of the sapphire supporting structure (Figure 3(c)) via sputtering. This stack included a 20-nm-thick titanium adhesion layer and a 200-nm-thick

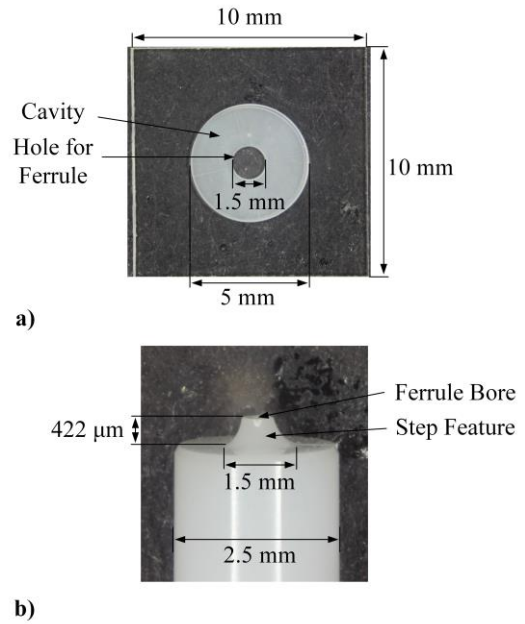


Figure 4: Stereoscopic images of the laser-micromachined (a) sapphire supporting structure and (b) zirconia ferrule.

platinum film serving as the bonding metal. A Dr. Sinter SPS-625 spark plasma sintering (SPS) press was used to provide the thermal and mechanical requirements for thermocompression bonding (Figure 3(d)). A schematic of the assembly inserted into the machine's chamber is shown in Figure 5.

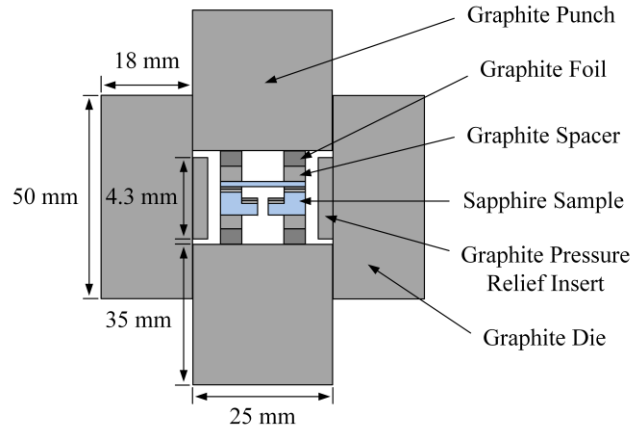


Figure 5: Schematic of the assembly that was inserted into the SPS press for thermocompression bonding.

The sapphire sample was sandwiched between graphite spacers and compressible graphite foil. Each spacer and piece of foil had a thickness of 1 mm and a 5 mm central hole to selectively apply pressure at the bonding interface. The graphite punches, which are housed within an annular graphite die, compress the foil until the outer edge of the punch reaches the annular graphite pressure relief insert. This setup, developed by Mills et al. [9], results in a fixed load on the sapphire sample based on the compressive strength of the foil. The thickness of the pressure relief insert was set to 4.3 mm to prevent diaphragm buckling. To bond the substrates, the temperature was increased and then held at 1000°C for one hour and

a pressure of ~ 5.3 MPa was applied by the punches. A stereoscopic image of the bonded sapphire sensing element is shown in Figure 6. The final fabrication step was the sputter deposition of a 20-nm-thick titanium adhesion layer and a 200-nm-thick platinum layer on the backside of the diaphragm for optical reflectivity (Figure 3(e)).

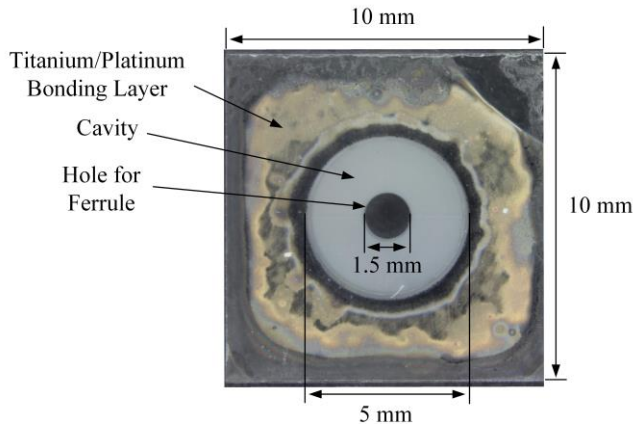


Figure 6: Stereoscopic image of the bonded sapphire sensing element.

Packaging

By using a stereoscope for alignment, a 70- μm -diameter sapphire fiber, terminated on one end with a fiber channel (FC) optical connector, was housed in the 81-83 μm bore of the zirconia ferrule and the bare end was positioned flush with the step feature. The fiber was attached to the ferrule using Cotronics ResbondTM 989F alumina-based adhesive. This same adhesive was utilized to attach the remaining sensor package components. The fiber-ferrule assembly was then packaged in a 304 stainless steel tube and attached to the sensing element using a flip-chip placement system. A 17-mm-diameter 304 stainless steel housing was used for integrating the sensing element with testing setups. An image of the packaged sensor is shown in Figure 7.

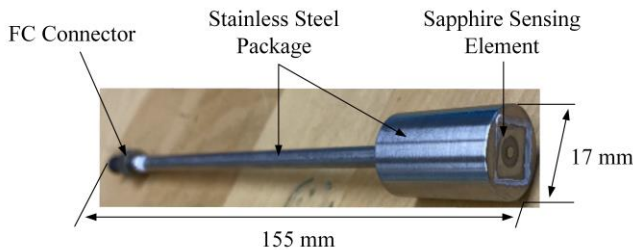


Figure 7: Image of the packaged sapphire optical pressure sensor.

CHARACTERIZATION

Frequency Response

The sapphire optical pressure sensor was calibrated in acoustic plane-wave tubes (PWT) at both ambient and elevated temperatures. Ambient calibration was performed in a PWT with an operating frequency range of 300-6700 Hz. A Brüel & Kjær 1/8" Type 4138-A-015 pressure-field microphone was used as the calibration reference. The sensitivity of the sapphire pressure sensor was measured by increasing the pressure from 140-160 dB SPL in increments of 5 dB SPL. A linear fit of the output voltage measurements yielded a sensitivity of 0.44 ± 0.02 $\mu\text{V}/\text{Pa}$. The frequency response was measured in 100 Hz increments at nominally 140 dB SPL. As shown in Figure 8, the sensitivity varied within $\pm 3\text{dB}$ throughout the tested frequency range.

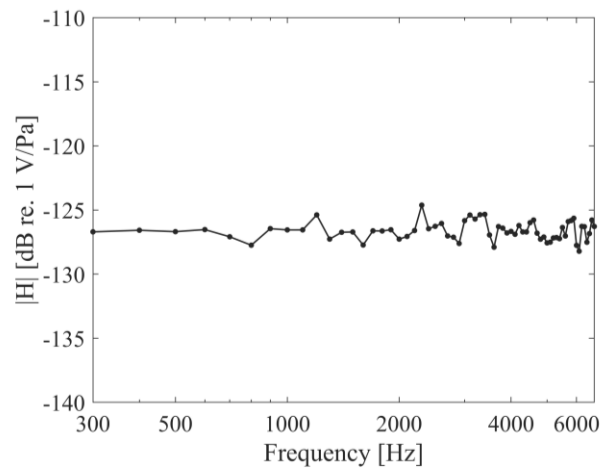


Figure 8: Measured frequency response of the sapphire optical pressure sensor at ambient conditions.

For high-temperature calibration, an alumina-based PWT with an operating frequency range of 300-2200 Hz was partially housed in a Thermo Scientific Lindberg/Blue M tube furnace. Due to the higher temperature requirement, a Brüel & Kjær Type 4182 probe microphone was used as the calibration reference. A 100-mm-long alumina tube was coupled to the end of the 100-mm-long steel tube provided by the manufacturer to increase the operating temperature of the probe microphone from 700°C to $>1000^\circ\text{C}$. The modified probe microphone was calibrated at ambient temperature to determine its frequency response.

The sapphire sensor, the probe microphone, and a Type K thermocouple were mounted in a Kovar plate sealed to the end of the PWT. Initially, the sensor was tested up to 500°C in 50°C increments, being brought back to ambient temperature at various points throughout. The sensor exhibited an unexpected order-of-magnitude increase in sensitivity upon reaching 500°C for the first time. It is believed this resulted from diaphragm buckling induced by a compressive stress generated from the constrained expansion of the 17-mm-diameter 304 stainless steel sensor package into the 17.1-mm-diameter machined hole in the Kovar plate. Measurements were repeated afterwards starting from ambient temperature to investigate repeatability of this change in response and the results for six tests are shown in Figure 9.

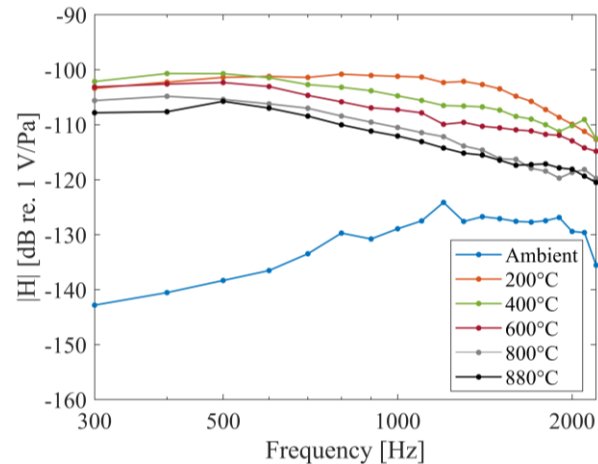


Figure 9: Measured frequency response of the sapphire optical pressure sensor from ambient temperature up to 880°C.

At ambient temperature, a low-frequency roll-on with a cutoff frequency of approximately 1100 Hz was present. This indicates the formation of a leakage path in the sensor package that attenuates its low-frequency response and was confirmed by a characterization of the sensor package that is detailed in the next section. Over an order-of-magnitude increase in sensitivity was measured at 200°C, which may be due to residual stress present from the initial compression of the diaphragm that resulted in an earlier onset of buckling. Post-buckled circular plates can exhibit over an order-of-magnitude increase in mechanical sensitivity near the critical buckling load and as the load is increased further it begins to decline [13]. This mechanical sensitivity trend agrees well with the remaining measured results at 400°C, 600°C, 800°C, and 880°C. The maximum tested temperature was limited by the experimental setup.

Package Characterization

A computerized tomography (CT) scan was performed on the packaged sensor to identify any leakage paths. A centerline cross section is shown in Figure 10(a) and a cross section offset just beyond the outer diameter of the zirconia ferrule is shown in Figure 10(b). As evidenced in Figure 10(b), a crack formed in the alumina-based adhesive that was used to seal the cavity, which resulted in the formation of an acoustic high-pass filter as shown in the ambient results of Figure 9. Once package characterization was complete, the sensor was tested at 900°C by adding additional insulation to the PWT; however, the sensing element fractured due to thermal stress.

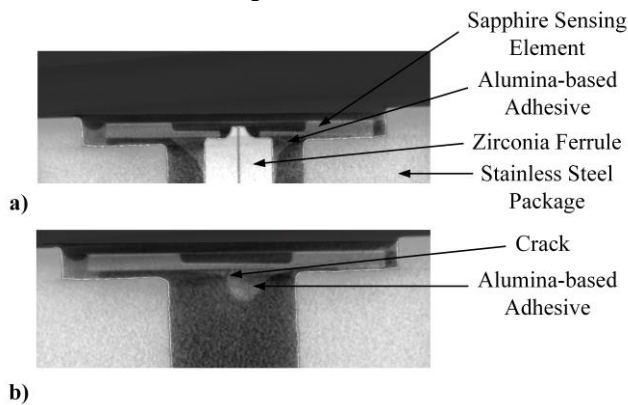


Figure 10: Cross-sectional CT-scan images of the packaged sensor (a) along the centerline and (b) offset just beyond the outer diameter of the zirconia ferrule.

CONCLUSIONS

The sapphire optical pressure sensor developed in this work demonstrated the ability to measure pressure fluctuations up to 880°C without the need for water-cooling or coupled probe tubes, both of which are utilized by commercially available sensors to operate at this temperature. Novel fabrication techniques, including ultrashort-pulse laser micromachining and thermocompression bonding via a SPS press, were implemented to realize unbuckled sapphire sensing elements. The packaged sapphire optical pressure sensor had a measured sensitivity of 0.44 $\mu\text{V}/\text{Pa}$ and a nominally constant response from 300-6700 Hz when initially tested in ambient conditions. High-temperature calibration up to 880°C revealed a structural breakdown of the alumina-based adhesive used to seal the cavity of the sensor. Furthermore, it is believed that a thermally induced compressive stress caused diaphragm buckling, resulting in a significant deviation from the predicted frequency response.

ACKNOWLEDGEMENTS

Financial support was provided by the NSF I/UCRC on Multifunctional Integrated System Technology (MIST) Center; IIP-1439644, IIP-1439680, IIP-1738752, IIP-1939009, IIP-1939050, and IIP-1939012. Additional financial support was provided by the Air Force Office of Scientific Research under award number FA9550-23-1-0519 (disclaimer: any opinions, findings, and conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the U.S. Department of Defense).

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