HIGH-TEMPERATURE ACOUSTIC EMISSION SENSING USING ALUMINUM NITRIDE SENSOR

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Abstract

We developed a heat-resistant acoustic emission (AE) sensor using AlN thin films and detected AE signals from molten-salt attack of Type-304 stainless steel pipe. Aluminum nitride (AlN) is a promising AE sensor element for high-temperature environment, such as gas turbines and other plants because AlN maintains its piezoelectricity up to 1200°C. Highly c-axis-oriented AlN thin-film sensor elements were prepared on silicon single crystals by rf magnetron sputtering. The AE sensor sensitivity increased with $d_{33}$ and thickness of AlN elements and the heat-resistant AE sensors were developed using AlN elements with high $d_{33}$ values and thickness of 9 μm. The sensor showed a constant sensitivity across a frequency range of 100 kHz to 1 MHz at ambient temperature. The sensor characteristics were evaluated at elevated temperatures up to 700°C. It was confirmed that the AE sensor works well up to 700°C and does not deteriorate. The AE sensor detected AE signals from frequent cracking of oxide scales produced by molten-salt attack (85% V$_2$O$_5$ + Na$_2$SO$_4$) at 700°C during cooling of the pipe.

Keywords: AE sensor, high temperature, aluminum nitride, molten-salt attack

Introduction

Plant monitoring systems are on high demand due to a series of recent accidents of power stations and chemical plants in Japan. Thus, there is the need to monitor high-temperature members in large structures. The AE method offers advantages such as observing the progress of plastic deformation and microscopic fracture in real time, locating a flaw using several AE sensors, and diagnosis capability without shutdowns. The AE method is used in a wide range of fields, including the inspection of manufactured products, monitoring the safety of structures and detecting natural disasters such as landslides [1]. The most common detection material for application in AE sensors is lead-zirconate titanate (PZT). Its Curie point, the temperature above which a piezoelectric material loses its piezoelectricity, is approximately 300°C. When an AE sensor with a PZT element is applied in high-temperature environment, a waveguide and/or a cooling device are essential and so it is difficult to measure AE correctly. Therefore, the development of a heat-resistant AE sensor that can be used above 300°C is strongly desired.

Aluminum nitride (AlN) is a piezoelectric material, which has a piezoelectric strain constant $d_{33}$ of 5.6 pm/V [2] and an ability to maintain piezoelectricity up to 1200°C [3]. AlN is a promising material as the active element of a heat-resistant AE sensor without a cooling device. However, the polarization of AlN cannot be controlled after preparation and sintered AlN poly-
crystals exhibit no piezoelectricity. This had been a major hindrance to the commercialization of AlN piezoelectric devices. After trying several methods, we finally succeeded in making highly oriented AlN thin films by rf magnetron sputtering [4].

In our previous studies [5-7], we developed both ordinary AE sensors and heat-resistant AE sensors using the AlN elements. The sensor characteristics were evaluated at ambient temperature and at elevated temperatures. To evaluate of $d_{33}$ and thickness of AlN elements on sensor sensitivity, AlN elements with $d_{33}$ from 2 to 7 pm/V and thickness from 3 to 9 μm were prepared. It is confirmed that the AE sensor sensitivity increased with $d_{33}$ and thickness of AlN elements [7]. The sensitivity of the heat-resistant AE sensor was also improved by a design of the sensor structure. The sensor characteristics were evaluated at elevated temperatures from 200 to 600°C. It was confirmed that the AE sensor works well at 600°C and does not deteriorate.

In this study, we developed a heat-resistant AlN AE sensor using AlN element of 9-μm thickness and Inconel-600 housing. The sensor characteristics were evaluated at elevated temperatures up to 700°C. Finally, using this AlN AE sensor, we succeeded in detecting AE signals from frequent cracking of the oxide scale produced by the molten salt attack (85% V₂O₅ + Na₂SO₄) at 700°C during cooling of the pipe.

**Experimental Procedures and Results**

*Preparation of AlN Thin Films*

AlN thin-film sensor elements were prepared on silicon single-crystal substrates (thickness: 0.625 mm) by rf magnetron sputtering. The sputtering conditions for the AlN thin film are reported in a previous study [4]. The thickness of AlN thin film was controlled by the sputtering time. The full width at half-maximum (FWHM) of the X-ray rocking curves of the film deposited under the optimized sputtering conditions was 2° and the film was highly oriented along the c-axis. A platinum (Pt) thin film used as the upper electrode was also prepared by rf magnetron sputtering. The crystal structure and orientation of the film were investigated by X-ray diffraction (XRD) analysis.

![Fig. 1 Diagram and photo of a heat-resistant AlN AE sensor.](image)

*Development of Heat-resistant AlN AE Sensors*

Disk-like AlN elements with 6-mm diameter were cut out from Si wafer for AE sensors. The $d_{33}$ value of each AlN element was measured using $d_{33}$ PiezoMeter System (Piezotest). The heat-resistant AlN AE sensors were developed using AlN element with high $d_{33}$ values and
thickness of 9 μm. The AlN elements were selected and directly fixed to the wear plate (alumina) by Ag paste just like an ordinary AE sensor. The diagram and the photo of the heat-resistant AlN AE sensor are shown in Fig. 1. The metal housing was made of Inconel 600, which has high thermal and corrosion resistances. A heat-resistant coaxial cable was developed for connecting the sensor to a preamplifier.

Evaluation of Characteristics of AlN AE Sensor

To measure the frequency responses of the AlN AE sensor, a commercial PZT AE sensor, AE-900S-WB (NF Corporation), was used to generate quasi-AE waves. This AE sensor is a wide-band model and possesses a constant sensitivity in the range of 100 kHz to 1 MHz (± 10 dB). A preamplifier (9913, NF Corporation) with a 40-dB gain in the frequency range of 100 Hz to 20 MHz was used for the AlN AE sensor. The AlN AE sensor was fixed to the PZT sensor using an acoustic coupler, Gelsonic (Nihon Kohden Corporation). Single-cycle sine wave (Vp-p: 20 V) was applied to the PZT sensor and the output from the AlN AE sensor was measured and expressed in dB (the ratio of the output to the input 20 V). The frequency of the sine wave was varied from 100 kHz to 1 MHz by 10 kHz intervals.

The frequency responses are shown in Fig. 2. It showed a constant sensitivity across the range of 100 kHz to 1 MHz (± 5 dB) like the PZT commercial AE sensor. While the PZT commercial AE sensor has a damper for constant wideband sensitivity, the AlN AE sensor shows a constant sensitivity without such a damper. The thickness of AlN element is 9 μm so that the resonance frequency must be much higher than 1 MHz. That is the reason why the AlN AE sensor shows a constant sensitivity, which is an advantageous feature of this heat-resistant AlN AE sensor.

![Fig. 2 Frequency responses of AlN AE sensor.](image)

Evaluation of AlN AE Sensor at High-temperatures

Figure 3 shows a diagram of the experimental setup for AE sensor responses at elevated temperatures. A stainless steel rod (diameter: 20 mm, length 400 mm) was used as an AE waveguide, which transmitted elastic waves from the PZT sensor outside an electric furnace. The waveguide was cooled by water outside the furnace and the temperature of PZT sensor was maintained under 60°C. The responses of the AlN AE sensor were measured at ambient temperature, 200, 300, 400, 500, 600 and 700°C after keeping the AlN AE sensor at each temperature for more
than 30 min. A tone-burst of 10 sine waves was generated and the output of AlN AE sensor was observed. A preamplifier was used for the AlN AE sensor.

The AlN AE sensor was coupled using an anti-seize paste (PBC® aka polybutylcuprysil, http://www.kspaul.de/TDB-eng/MP-PBC-engl.pdf). This is semi-synthetic grease with metallic powders and inorganic thickener. After high temperature exposure, it became powdery, but still functioned as a couplant during cooling. The sensor was mechanically held with a ceramic spring.

Figure 4 shows a generated envelope of a tone-burst wave with 10-cycle sine waves of 200 kHz, and the response signals of the AlN AE sensor at 300, 500 and 700ºC. It is confirmed that the sensitivity of the AlN AE sensor is maintained from ambient temperature up to 700ºC.

Fig. 4 Generated tone-burst wave (a) with 10 sine waves of 200 kHz and the response signals of AlN AE sensor at (b) 300º, (c) 500º and (d) 700ºC.
AE Monitoring of Molten Salt Attack by AlN AE Sensor

Accelerated oxidation of heat-resistant alloys by molten salt is well known as the most dangerous damage, and often called as catastrophic oxidation. We measured AE signals by molten-salt attack (85 mol% V_2O_5 + Na_2SO_4) by using the AlN AE sensor. We used a similar experimental setup reported previously [8]. Figure 5 shows an experimental setup for molten-salt attack and AE monitoring method of a hot stainless steel pipe (type-304) of 34-mm diameter, 3.0-mm thickness and 1200-mm length. The PZT sensor (AE-900S-WB) was also mounted as a reference on the pipe end, where the temperature was lower than 40°C. Preamplifiers (2/4/6, PAC) with 40-dB gain were used for the AlN AE and PZT sensor. We used an AE monitoring system (PAC, DSP PCI-2). We placed 0.1 g of a mixed salt (85 mol% V_2O_5+Na_2SO_4) on the upper portion of the pipe at the center of the furnace and then heated the pipe.

Figure 6 shows cumulative AE hits of the AlN sensor and temperature of the pipe in the furnace. The pipe was heated to 700°C and kept for 3 hours, then the power of the furnace was turned off and the pipe was allowed to cool. The threshold for AE hits of the AlN AE sensor was set at 10 mV. We detected few AE hits during heating and holding, but monitored frequent AE hits during cooling. The similar AE during cooling was observed in a previous study using optical fiber sensors [8]. Therefore, the AE hits must be caused by frequent cracking of the oxide.
Fig. 7 Waveform of AE detected by AlN AE sensor.

Fig. 8 FFT of waveform in Fig. 7.

Fig. 9 Same signal as Fig. 7 by PZT sensor.
scales (and possibly salt cracks at lower temperatures) produced by the molten-salt attack during cooling of the pipe. A typical AE waveform is shown in Fig. 7. The power spectra of the AE (Fig. 8) showed many peaks below 100 kHz and a few peaks around 110, 240 and 360 kHz. The latter two appear to be due to F(1,2) and F(2,2) cylindrical waves from the cracking sources propagating on the pipe [9]. When the same signal (Fig. 7) was detected by the PZT sensor outside the furnace, the dispersion of cylindrical waves stretched the signal by several times, as shown in Fig. 9.

We have not tested for the durability on this AlN AE sensor, but it worked at 700°C for at least several hours for these experiments. The high-temperature couplant remained effective. It is confirmed that the AlN AE sensor detected AE signals from the fracture of the oxide scales produced by the molten-salt attack at 700°C during cooling of the pipe.

Summary

We developed a heat-resistant AE sensor using AlN thin films and detected AE signals from molten-salt attack of Type 304 stainless steel pipe. Aluminum nitride (AlN) is a promising acoustic emission (AE) sensor element for high-temperature environments such as gas turbines and other plants because AlN maintains its piezoelectricity up to 1200°C. Highly c-axis-oriented AlN thin-film sensor elements were prepared on silicon single crystals by rf magnetron sputtering. The heat-resistant AE sensors were developed using AlN elements with high $d_{33}$ values and thickness of 9 μm. The sensor showed a constant sensitivity across a wide band of frequencies in the range of 100 kHz to 1 MHz at an ambient temperature. The sensor characteristics were evaluated at elevated temperatures up to 700°C. It was confirmed that the AE sensor works well up to 700°C and does not deteriorate for at least several hours. The AE sensor detected AE signals from frequent fractures of the oxide scales produced by the molten salt attack (85% V$_2$O$_5$ + Na$_2$SO$_4$) at 700°C during the cooling of the pipe.

Acknowledgment

A part of this study was supported by Industrial Technology Research Grant Program in 2004 from New Energy and Industrial Technology Development Organization (NEDO) of Japan.

References


