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Measurement of output voltage of aluminum nitride thin film as a pressure sensor at high temperature

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Abstract

The output voltage of sensor elements using AlN thin film was investigated at temperature up to 973 K with the frequency of 1 and 10 Hz under the stress of 200 ± 40 N, respectively, in nitrogen atmosphere. The output signals were ascertained at temperatures up to 937 K with both frequencies. The intensity of the output voltage before heating was 1.4–2.2 V approximately. It could be held close to the initial level up to 673 K, however, it declined over 773 K and came down to less than 10% of the original level at 873 and 973 K in the case of SUS 304 electrodes because of the degradation of stainless steel. In the case of Inconel 600 electrodes, it was approximately constant up to 873 K. However, the sample showed anomalously high output voltage of more than 13 V by the fluttering of the testing machine at 973 K. In spite of the exceeding condition, the elements responded the applied load. It is considered that a sensor element using AlN thin film can be applied as a pressure sensor at temperature up to 973 K.

Keywords: Films; Piezoelectric properties; Nitrides; Sensors; AlN

1. Introduction

The reduction of CO_2 gas emission is urgent from the view point of countermeasure against global warming. To reduce CO_2 gas emission, the lean burn engine has become of interest in recent years, not only for improvement of fuel efficiency, but also for operation with various kinds of fuels.[1] However, the combustion in lean burn engines is unstable and it is difficult to optimize the driving conditions, e.g. fuel–air mixing ratio. Nagata and Harashima have reported that the microcomputer control using data from a $PbZrO_3{\cdot}PbTiO_3$ (PZT) pressure sensor is effective to obtain stable driving of engines.[2] However, they have not described the details of their experiments, e.g. how to set the sensor element in the engine. It is not sure, but, from their objective temperature rage of −30 to 180 °C, it can be assumed that they have not measured the pressure inside a cylinder directly. To obtain more stable combustion condition and improved fuel efficiency, in situ measurement of the combustion pressure inside the cylinders of an engine is promising. In such case, the required heat resistance for the sensor elements may be more than 700 K. And so, conventional piezoelectric ceramics such as PZT cannot to be applied because of their Curie point lower than the temperature in the cylinders of engines. Therefore, high heat resistant sensor elements with low price are required.

In this respect, aluminum nitride (AlN) thin film is of interest, because of its piezoelectric property at high temperature up to 1150 °C.[3] The piezoelectric property of AlN thin film originates from its oriented crystalline nature and so, it has no Curie point. Akiyama et al. have reported that they have obtained highly orientated AlN thin film by the plasma sputtering method and shown its possibility to be applied as sensors elements at high temperature.[4–6] Stubbs and Dutton reported that AlN thin film could be applied as an ultrasonic sensor at high temperature up to 900 °C.[7] However, the application of AlN thin film as a pressure sensor at high temperature, e.g. for controlling an engine, is still not reported. This article describes a preliminary measurement of the output voltage of AlN thin film at high temperature up to

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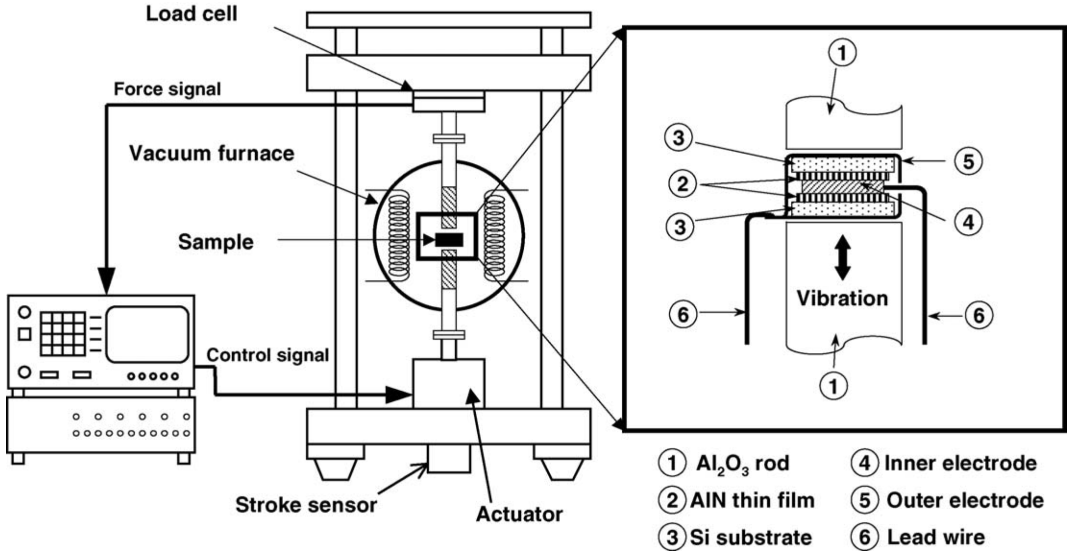

Fig. 1. Schematic illustration of experimental setup for the measurement of output voltage of AlN thin films.

973 K, in order to confirm the possibility of its application to in situ measurement of combustion pressure of engines.

2. Experimental procedure

The measurements of output voltage of AlN thin film were performed using the experimental setup illustrated schematically in Fig. 1. The AlN thin films were formed by the previously reported procedure[4,5] on single crystal Si substrate with the size of 17 mm × 17 mm × 0.5 mm. The vibration load was applied to the sample, which is assembled as shown in Fig. 2, using the fatigue testing machine (base model is Micro-servo MMT-500NB-10, Shimazu Co. Ltd., Kyoto, Japan, the maximum load capacity of 500 N and the maximum frequency of 100 Hz). A plate and a foil of stainless steel (SUS 304) and nickel based alloy (Inconel 600) are used as the electrodes material respectively as shown in Fig. 2. The output voltage was measured by a storage oscilloscope via the charge amplifier (AG 2101, NEC-Sanei Co. Ltd., Tokyo, Japan). The measurement was carried out for each 100° from room temperature up to 973 K with the frequency of 1 Hz and 10 Hz under the stress of 200 ± 40 N, respectively, in nitrogen atmosphere. The AlN thin film elements and electrodes were observed with an optical microscope after the measurements. In order to confirm the microstructural change of electrodes materials, the SUS 304 and Inconel 600 foils with the size of 15 mm × 15 mm were observed by a scan-

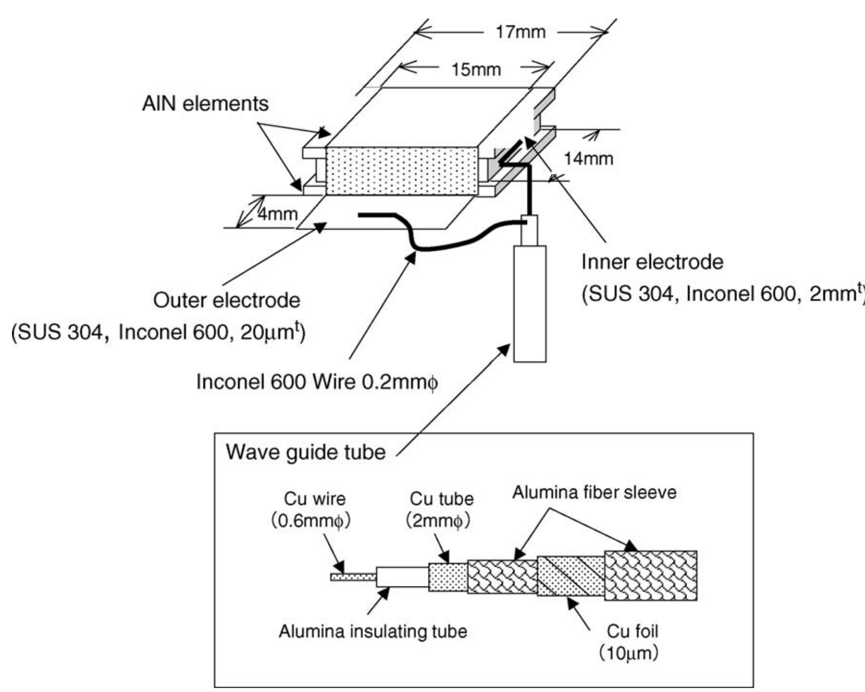

Fig. 2. Schematic illustration of assembly of a sample.

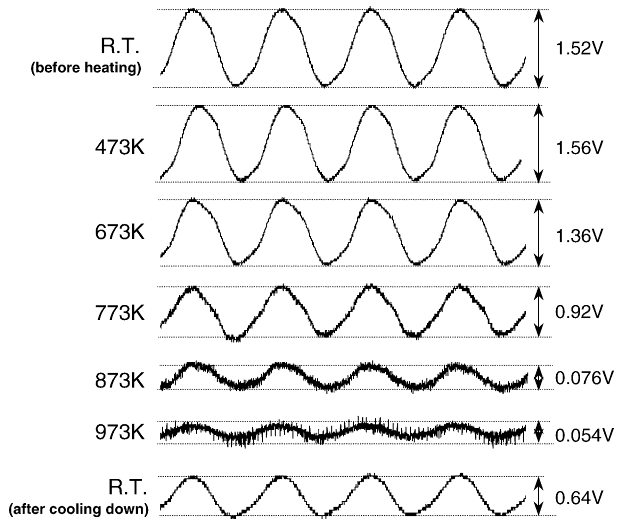

Fig. 3. Change of profiles of output voltage with the frequency of 1 Hz by the oscilloscope with elevated temperature (SUS 304 electrodes).

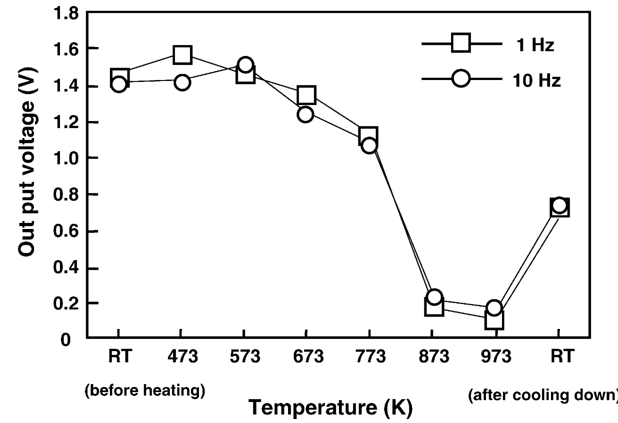

Fig. 5. Change of output voltage as a function of temperature.

ning laser microscope (VK-8500, Keyence Co. Ltd., Osaka, Japan) after heat treatment in nitrogen atmosphere at 673, 773 and 873 K for 30 min.

3. Results and discussions

Figs. 3 and 4 showed the profiles of output voltage at 1 Hz and 10 Hz observed by oscilloscope with increasing temperature in the case of SUS 304. The output signals were ascertained at temperature up to 937 K with both frequencies. Fig. 5 showed the change of output voltage as a function of increasing temperature. The intensity of the output voltage before heating was 1.4–1.5 V approximately and that could be held close to the initial level up to 673 K. However, it declined over 773 K and came down to less than 10% of the original level at 873 and 973 K in the last result. After cooling down as it was, it recovered to about 50% of the original level. A newly assembled sample using the same elements, showed output voltage of 1.32 V.

Low-amplitude peaks were observed over 773 K as shown in the figures. Since the frequency of these peaks was 60 Hz, these were assumed to be due to the noise from the electric furnace after increasing its generating power. All profiles looked to be distorted sine curve shapes as show in the figures. However, since output signals from the load cell by which the fatigue testing machine was controlled, were also very similarly distorted, this was probably due to the mechanical problems, e.g. the less smooth moving at the part of a screw clamp. Therefore, it is considered that the elements using AlN thin film appropriately reflected the applied pressure.

Fig. 6 showed the profiles of output voltage at 10 Hz with increasing temperature in the case of Inconel 600. The profiles looked to be more distorted shape. However, the sample showed larger output voltage of 2.2–2.5 V than that of SUS 304 until 873 K. Since the output signal was more distorted and the output voltage showed anomalously high value of more than 13 V at 973 K by the fluttering of testing machine, it is not shown

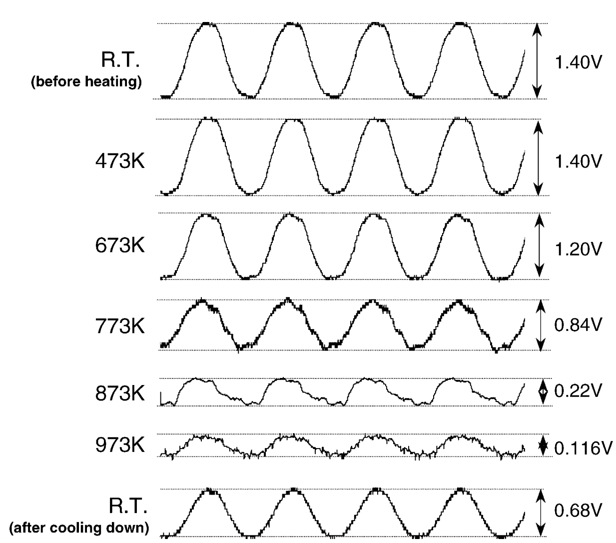

Fig. 4. Change of profiles of output voltage with the frequency of 10 Hz by the oscilloscope with elevated temperature (SUS 304 electrodes).

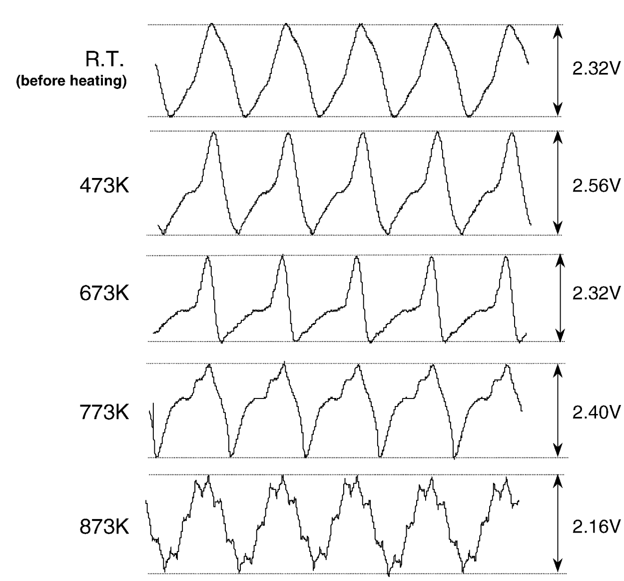

Fig. 6. Change of profiles of output voltage with the frequency of 10 Hz by the oscilloscope with elevated temperature (Inconel 600 electrodes).

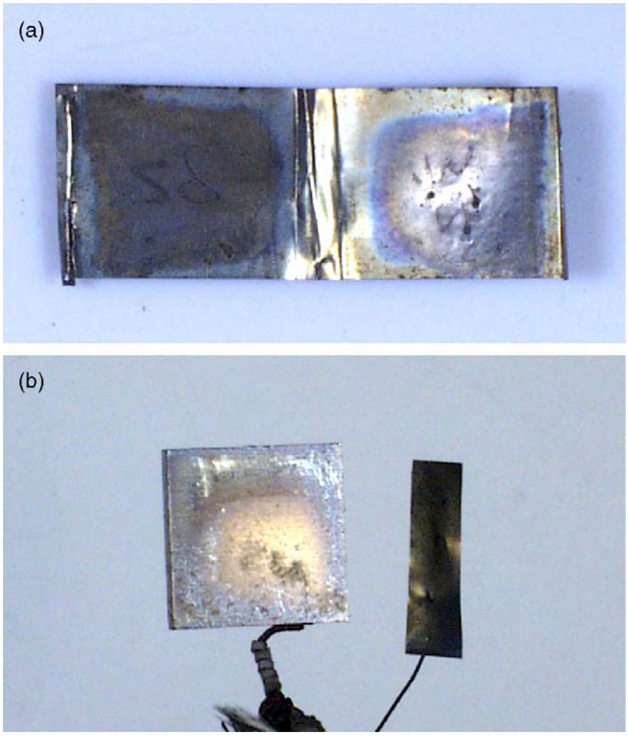

Fig. 7. Appearance of SUS 304 electrodes after measurement at 973 K: (a) outer electrode, (b) inner electrode.

in Fig. 6. However, it is considered that the elements exactly responded the applied cyclic load at 973 K.

Figs. 7 and 8 showed the appearance of both electrodes of SUS 304 and Inconel 600 after the measurement, respectively. In the case of SUS 304, according to the change of the color of both electrodes, it could be seen that they were oxidized with residual oxygen. On the other hand, in the case of Inconel 600, metallic luster can be observed at the inner electrode and inside

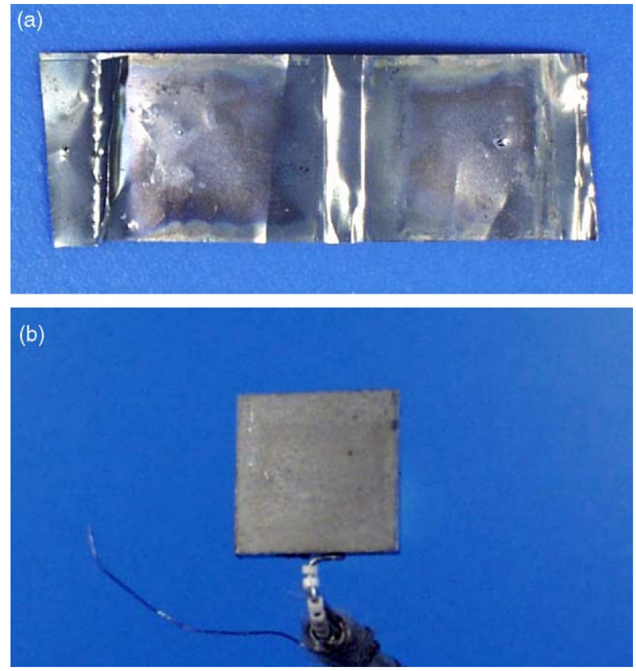

Fig. 8. Appearance of Inconel 600 electrodes after measurement at 973 K: (a) outer electrode, (b) inner electrode.

of the outer electrode even after heating up to 973 K. It was reported that stainless steel is degraded by heating over 500 °C because of the deposition of carbide at the grain boundary.[8] Fig. 9 showed the change of surfaces of the heat treated SUS 304 foils as scanning laser micrographs. Since black grains come to be clearly observed from 773 K, and the size of the grains became more than 10 μm at 873 K, it is considered that, these black grains penetrated the foil, if these black grains formed from both sides of a foil. Thus, according to the degradation of SUS 304 electrodes, the output voltage also assumed to be decreased.

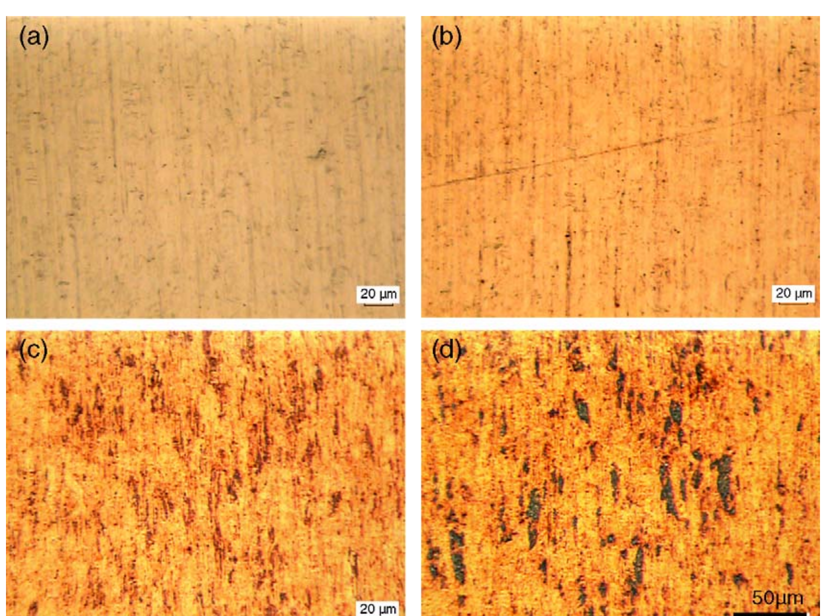

Fig. 9. Change of the surfaces of SUS 304 foil with heat treatment for 30 min: (a) before heating, (b) 673 K, (c) 773 K, (d) 873 K.

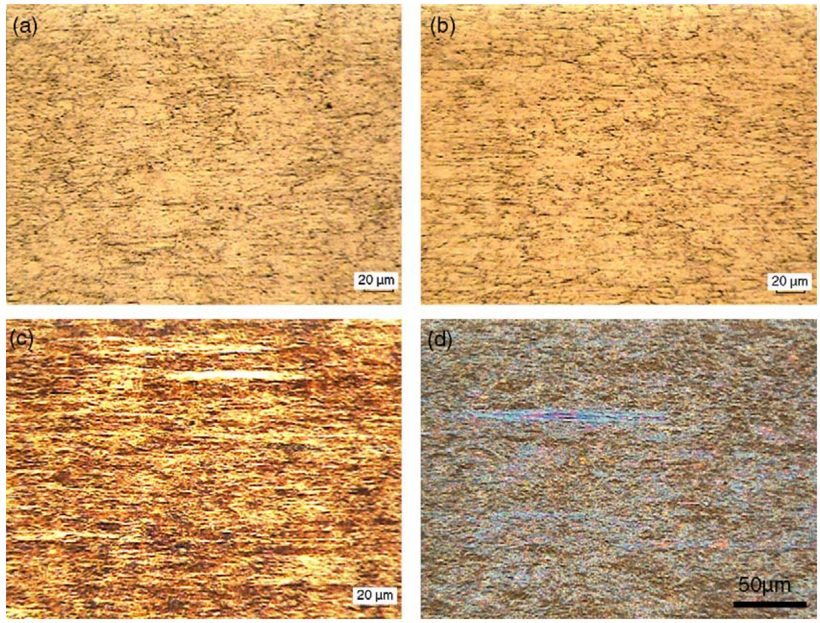

Fig. 10. Change of the surfaces of Inconel 600 foil with heat treatment for 30 min: (a) before heating, (b) 673 K, (c) 773 K, (d) 873 K.

On the contrary, in the case of the Inconel 600 foils, in spite of the color change due to oxidation, no deposited grains observed, as shown in Fig. 10. It is considered that the most frequent cause of the decrease in output voltage using SUS electrodes is the degradation of the electrodes.

Fig. 11 showed an optical micrograph of the surface of an element after the measurement. Although the part of Si substrate near the edge was oxidized, the AlN thin film and surrounding part of Si substrate revealed no change as shown in Fig. 11. It is considered that both electrodes played a role of oxygen getter.

In spite of the degraded SUS 304 electrodes, the output voltage of the element using AlN thin film was recovered about 50% of the initial level. And the output voltage of a newly assembled sample using the same elements was almost same as that before heating. The remaining degraded output voltage of 50% may be due to experimental failings, e.g. thermal expansion mismatch between the electrodes and the elements. This is also the reason why the fluttering of testing machine at 973 K in case of Inconel electrodes conceivably. Therefore it is considered that the original property of AlN thin film is distinctly less degraded by heating up to 973 K.

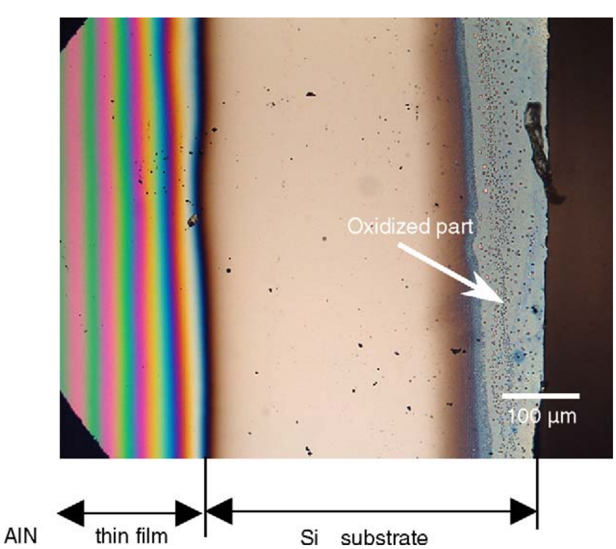

Fig. 11. Optical micrograph of the surface of the element using AlN thin film.

4. Conclusion

The output voltage of a sensor element using AlN thin film was measured at the temperature up to 973 K using SUS 304 and Inconel 600 electrodes, in order to confirm the possibility of its application as a high temperature pressure sensor. In the case of SUS 304, the output signals were ascertained at the temperature up to 937 K. The intensity of the output voltage could be held close to the initial level up to 673 K, however, it declined over 773 K, because of the degradation of electrodes. Since it recovered to the initial level in a newly assembled sample using the same elements, the elements were little degraded by heating up to 973 K. In the case using Inconel 600 electrodes, the profile of the signals was more distorted than that using SUS 304. However, the intensity of the output voltage was almost same as that of the initial level up to 873 K and the elements could respond applied load up to 973 K. It can be concluded that the sensor element using AlN thin film can be applied as a high temperature pressure sensor.

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