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Pressure Sensing Properties of AlN Thin Films Sputtered at Room Temperature

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Abstract

Aluminum nitride (AlN) thin films with a TiN buffer layer have been fabricated on SUS430 substrate by RF reactive magnetron sputtering at room temperature under 25~75% N_2 /Ar. The characterization of film properties were performed using surface profiler, X-ray diffraction, X-ray photoelectron spectroscopy(XPS), and pressure-voltage measurement system. The deposition rates of AlN films were decreased with increasing the N_2 concentration owing to lower mass of nitrogen ions than Ar. The as-deposited AlN films showed crystalline phase, and with increasing the N_2 concentration, the peak of AlN(100) plane and the crystallinity became weak. Any change in the preferential orientation of the as-deposited AlN films was not observed within our N_2 concentration range. But in the case of 50% N_2 /Ar condition, the peak of (002) plane, which is determinant in pressure sensing properties, appeared. XPS depth profiling of AlN/TiN/SUS430 revealed Al/N ratio was close to stoichiometric value (45:47) when deposited under 50% N_2/Ar atmosphere at room temperature. The output signal voltage of AlN sensor showed a linear behavior between 26~85 mV, and the pressure-sensing sensitivity was calculated as 7 mV/MPa.

Keywords: Piezoelectric, AlN thin film, Pressure sensor, RF reactive sputtering, Room temperature deposition

1. INTRODUCTION

Over the past decades, pressure sensors such as Si piezoresistive semiconductor based on Si MEMS technology are the most prevalent type of pressure sensor because of its easy integration, high sensitivity, highly befitting for mass production, and they dominate over 83% of the market share of the pressure sensor [1]. However, its operating temperature has been limited below 573 K, approximately. Therefore an application to combustion pressure sensor for automobile engine such as gasoline direction injection (GDI) type and common rail type, which needs high temperature more than 673 K and high pressure more than 350 MPa, is limited.

Piezoelectric materials have attracted much attention due to their promising properties. Among these materials, Aluminum nitride (AlN) thin film has been widely studied because of its excellent characteristic, which includes the following: the AlN thin film has a high melting point (2673 K), a high energy band gap (6.2 eV), good thermal conductivity (260 Wm^1K^1), a rapid acoustic velocity (6000 m/s), extreme hardness, a low acoustic impedance, and good chemical stabilities [2,3]. Especially, AlN thin film is of interest, because of its piezoelectric property at high temperature up to 1423 K [4]. The piezoelectric property of AlN thin film originates from its oriented crystalline and it has no Curie point. AlN has Wurtzite structure, and the Al-N bond length of the c-axis (0.498 nm) is longer than that of the a-axis (0.311 nm). The piezoelectricity of AlN ($d_{33=}$~7pC/N) is originated from the polarization difference in the c-axis direction, and the polarization of AlN is maintained to about 1423 K.

Due to the c-axis orientation tendency of AlN thin film deposited on any substrate materials, the deposition of AlN in thin film type has been carried out by various methods, such as physical vapor deposition, chemical vapor deposition, and molecular beam epitaxy [5-10]. However, techniques such as CVD and MBE require high substrate temperature of above 1273 K compared with PVD. Thus, PVD methods such as reactive magnetron sputtering are frequently used for the deposition of AlN film because the substrate temperature can be lowered even to room temperature.

Akiyama *et al*. have reported that they have obtained highly

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oriented AlN thin film by sputtering method at 473~673 K and shown its possibility as sensor elements at high temperature [11-13].

In this study, we report on the pressure sensing properties of AlN thin films deposited by RF reactive magnetron sputtering at room temperature.

2. EXPERIMENTAL

2.1 Deposition of thin films

Thin films were deposited by RF reactive magnetron sputtering (LSS-01, J&L Tech, Korea) from 5N-grade metal target (Dia. 3") under various ratio of N_2 and Ar onto disc-type SUS430 substrate (Dia. 17 mm) at room temperature. The SUS430 substrates were washed with acetone, methanol and rinsed in water for 15 minutes by ultrasonic cleaner before deposition in order to remove contaminants. Pre-sputtering was performed for 10 minutes to remove any contamination on the target. The base vacuum level of deposition chamber was lower than 0.9 mPa,

In our preliminary experiments, it was confirmed that direct deposition of AlN films to SUS430 resulted in excessive stress and resultant cracks in the AlN films owing to the large difference in thermal expansion coefficients between SUS430 (~10.4 ppm/K) and AlN (4.9 ppm/K). To avoid the cracks in the AlN thin films, it is necessary to insert buffer layer, which has the intermediate thermal expansion coefficient between SUS430 as a substrate and AlN. And the buffer layer must be conductive in order to use as an electrode. Titanium nitride (TiN), which has an appropriate thermal expansion coefficient (~9.4 ppm/K) and metallic property, was selected as the buffer layer. TiN films with a thickness of ~400 nm were deposited under 25% N_2/Ar at room temperature. After the deposition of TiN as a buffer layer, 150 nm-thick AlN films were deposited onto the TiN buffer layer. The N_2/Ar ratio in the chamber was varied to 25~75% at room temperature.

The specific deposition conditions of TiN and AlN films are shown in Table 1.

Table 1. Deposition conditions of TiN and AlN

Item	TiN	AlN
Target	Ti	Al
Power	3	2
Working Pressure[Pa]	0.6	0.3
Atmosphere [%]	25% N_2/Ar	25~75% N_2/Ar
Substrate temp. [K]	R.T.	R.T.

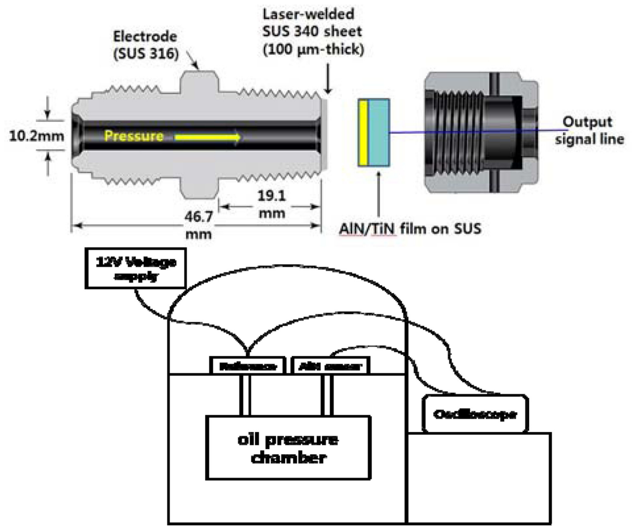

Fig. 1. Cross section of pressure-sensing module and schematic diagram of oil pressure application apparatus.

2.1.1 Characterization

Thickness and deposition rate of AlN thin films were measured with a surface profiler (Alpha step 500, Tencor, USA). Crystalline phases of the films were confirmed using X-ray diffraction at 10o to 70o (Rigaku DMAX2200, Japan, Cu Ká, 40 kV/30 mA). The depth profiling along to the deposition direction was performed using XPS.

The evaluation of pressure-sensing properties was performed using an oil pressure application apparatus with a reference sensor (UNIK5000, GE, USA), whose output signal range was 0~5 V with the accuracy of ±0.04% FS. The AlN-coated SUS430 discs were implemented into pressure-sensing modules. We sandwiched Cu foil (dia=13 mm) as the top electrodes between AlN thin film and laser-welded SUS sheet, and the modules were installed into pressure application apparatus as depicted in Fig.1. The range of the applied pressure was 1~10 MPa with a frequency of 2 Hz.

3. RESULTS AND DISCUSSIONS

The deposition rates of AlN films deposited by RF reactive magnetron sputter were decreased from 1.8 nm/min to 1.1 nm/min with increasing the N_2 concentration from 25% to 75% in the deposition chamber. This result can be explained by the difference in the bombardment energy of Argon and Nitrogen ions based on the difference in the mass of Argon and Nitrogen ions to bombard Al target. The mass of Ar (molecular weight 40) is heavier than that of N_2(molecular weight 14), and the resultant sputtering yield

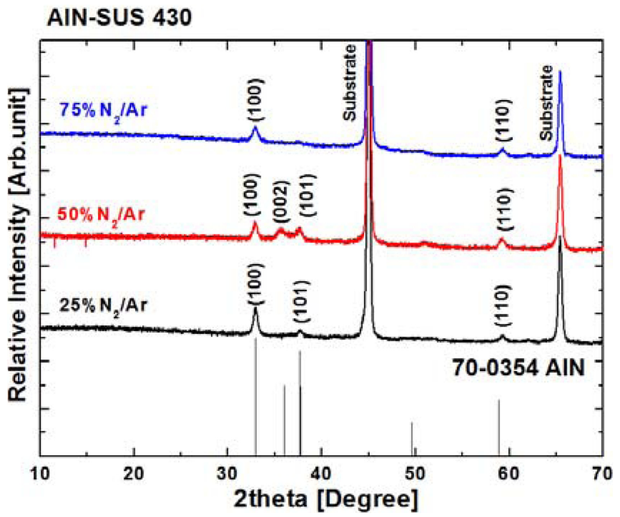

Fig. 2. XRD patterns of AlN thin films on SUS430 at various N_2 /Ar ratio.

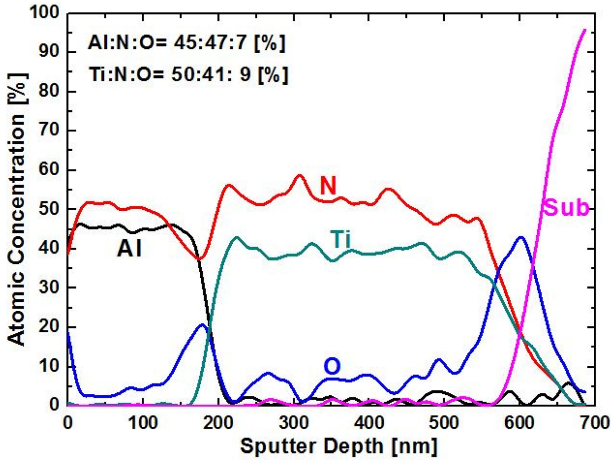

Fig. 3. XPS depth profiling of AlN/TiN/SUS430 deposited under 50% N_2/Ar atmosphere at room temperature.

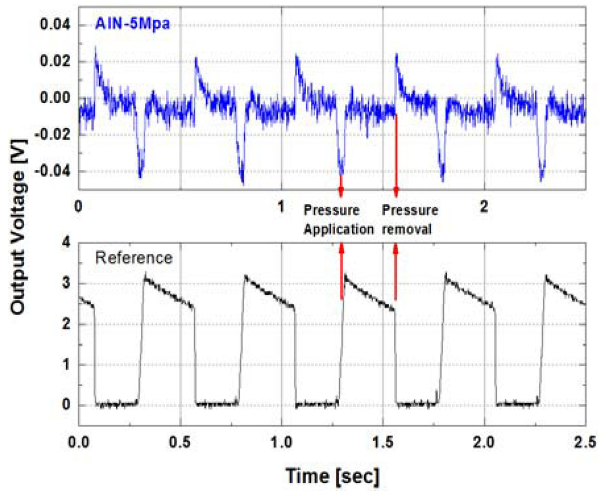

Fig. 4. Pressure sensing behavior of AlN sensor module composed of AlN thin films (@ 5 MPa).

of Ar is higher than those of that of Nitrogen ion. Therefore, the increase in the N_2 concentration results in the decrease in Al sputtering yield. In addition, a nitride reaction of the target surface was increased with increasing N_2 concentration, and the sputtering yield of the target was decreased with increasing the nitride on the target surface. The binding energies of AlN and Al are 9.1 eV, and 3.35 eV, respectively. Thus the sputtering yield of AlN is lower than that of Al [14].

Fig. 2 shows X-ray diffraction (XRD) patterns of AlN thin films deposited at room temperature under different N_2 concentrations. As-deposited AlN films showed crystalline phase, and with increasing the N_2 concentration, the peak of AlN(100) plane and the crystallinity became weak. The peaks of (100), (101) planes were observed at the 25% N_2 concentration. With increasing N_2 concentration to 50%, the peak of (002) plane appeared. At the 75% N_2 concentration, the peak of (002) plane was decreased. These results can be explained by the reaching particle energy on the growth surface and the difference of the Al, N composition. The AlN thin film at 25% N_2/Ar ratio is grown with (100) planes owing to the excess of Al atom by high sputtering yield. At 75% N_2/Ar ratio, the intensity of (002) plane was decreased by the excess of nitrogen gas and a low kinetic energy of the reaction particles. In the high N_2 concentration, the kinetic energy of the sputtered particles is low because kinetic energy of Ar is mainly used as the generating and accelerating of the secondary electrons by the accumulation of target nitrification [15-17]. So, it is thought to be appropriate that AlN thin films are deposited in N_2 gas concentration of in the region 50% approximately [18].

Fig. 3 shows the depth profiling by XPS along to the deposition direction. The XPS depth profiling of AlN thin film with a TiN buffer layer on SUS430 substrate revealed Al/N ratio was close to stoichiometric value (45:47) when deposited under 50% N_2/Ar atmosphere at room temperature. Oxygen component is observed from surface to about 30 nm in depth, and somewhat high ratio of oxygen in the boundary of AlN/TiN and TiN/substrate is thought to be caused by target exchange. Ti/N ratio showed Ti excess (50:41) Titanium has similar thermal expansion coefficient (~8.6 ppm/K) and electrical conductivity to TiN. Therefore, it is thought that there are little effects on the properties of AlN films.

Fig. 4 shows the pressure sensing behavior of AlN sensor module deposited under 50% N_2/Ar atmosphere. The applied pressure was 5 MPa with a frequency of 2 Hz. The output signal voltage of the reference sensor showed about 3 V, and that of AlN

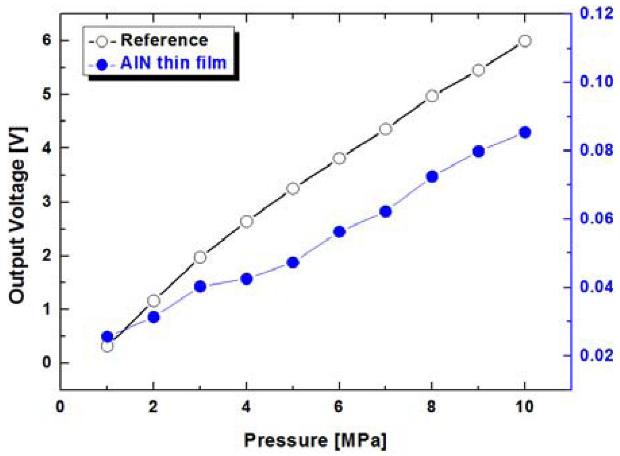

Fig. 6. Pressure sensing characteristics of AlN sensor (50% N_2/Ar).

sensor module appears at the moment of pressure application and removal with a pulse pattern. Unlike Si piezo-resistive sensor, the piezoelectric properties of AlN are produced by a change in the polarization in the c-axis direction originated from a change in the Al-N bond length. Therefore, the pressure-induced output voltage of AlN thin film appears at the moment of pressure application and removal. Electrical charge of dipole of AlN is changed when the dipole shrink (pressure application) and expand (pressure removal). The output signal voltage showed about 43 mV with a rapid-respond and reproducible pattern.

If we consider the output signal of commercial reference sensor is generated through a noise-cut and amplifying circuit, the output signal of AlN thin film sensor fabricated in this work without any circuit shows very distinct and reproducible output signal voltage, and a possibility as a pressure sensing element for high temperature and high pressure use.

Fig. 5 shows pressure sensing characteristics of a commercial reference sensor and AlN sensor fabricated in this work in the pressure range 1~10 MPa. The output signal of AlN sensor showed a linear behavior between 26~85 mV, and that of the reference sensor showed 0.3~6 V. The pressure-sensing sensitivities of AlN and reference sensor were calculated as 7 mV/MPa and 0.6 V/Mpa, respectively.

4. CONCLUSIONS

In this research, aluminum nitride (AlN) thin films with a TiN buffer layer have been fabricated on SUS430 substrate by RF reactive magnetron sputtering at room temperature under 25~75% N_2 /Ar. The deposition rates of AlN films were decreased from 1.8 nm/min to 1.1 nm/min with increasing the N_2 concentration owing to the lower mass of nitrogen ions than Ar. The as-deposited AlN films showed crystalline phase, and increase in the N_2 concentration resulted in the weak crystallinity. Any change in the preferential orientation of the as-deposited AlN films was not observed within our N_2 concentration range. But in the case of 50% N_2/Ar condition, the peak of (002) plane, which is determinant in pressure sensing properties, appeared. XPS depth profiling of AlN/TiN/SUS430 revealed Al/N ratio was close to stoichiometric value (45:47) when deposited under 50% N_2/Ar atmosphere at room temperature. The output signal voltage of AlN sensor showed a linear behavior between 26~85 mV, and the pressure-sensing sensitivity was calculated as 7 mV/MPa.

The output signal of AlN thin film sensor fabricated in this work showed very distinct and reproducible output signal voltage, and a possibility as a pressure sensing element for high temperature and high pressure use.

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