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Study of Force-feedback Tactile Sensor Fabricated by AlN Doped ZnO Piezoelectric Film

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Keywords：Tactile sensor, Sol-gel, Thin film, ZnO, AlN

ABSTRACT

In this study, we have developed a flexible AlN doped ZnO tactile sensor. This flexible sensor can be applied on the complex surface of the device. The tactile sensor is fabricated by AlN doped ZnO thin film deposited on the flexible polyimide (PI) substrate or copper substrate. The sol-gel method and spin-coating method are utilized to prepare the AlN doped ZnO piezoelectric films. We have also investigated the influences of the speed of spin-coating on the crystallinity and piezoelectric properties of the thin films. The methods of X-ray diffraction and scanning electron microscopy are used to examine the crystallinity and the surface topography of the AlN doped ZnO thin films. In addition, the force-feedback analyses are performed to compare the output voltages between the sensors on the PI substrate and copper substrate. The response of force feedback proves the availability of this flexible tactile sensor and its robust piezoelectric properties.

INTRODUCTION

The piezoelectricity was first discovered in 1880 by Pierre Curie and Jacques Curie. They found the electrical potential difference as the mechanical stress was applied on the quartz and the voltage was proportional to the stress (Curie and Curie, 1880). Therefore, the piezoelectric effect could be understood about the linear electromechanical interaction between the mechanical and the electrical state. Paul Langevin in 1917 developed transmitters and receivers for underwater applications by a quartz crystal (Bjorno, 2003). Then, the piezoelectric materials were widely used in the following decades (Mason, 1981). Nowadays, the flexible electronics have attracted much research attention. The flexible substrates, films, and fibers have the advantages compared to semiconductor materials in terms of the cost, large scale, fabrication and biocompatibility. These diverse substrate materials have been investigated for the displays, memory, circuitry, photovoltaic devices and MEMS sensors (Chen et al., 2014; Liu et al., 2008; Shih et al., 2011). In this study, we fabricate a tactile sensor based on the thin-film technology, which was commonly used in the transduced techniques, including the capacitive component, pizoresistor, thermoresistor, inductor, piezoelectric film, and magnetic devices (Fu and Li, 2015; Lin et al., 2013; Chou et al., 2015). In addition, the tactile sensor can be further applied to the minimally invasive surgery for improving the tactile sensing capability of instruments (Eltaib and Hewit, 2003). Zinc oxide (ZnO) has a crystal structure with the center symmetry, but no axis symmetry. This specific lattice symmetry results in a piezoelectricity and pyroelectricity. ZnO is also a transparent and lead-free piezoelectric material, and has been largely studied in the literatures during the past decades (Malucelli et al., 2017; Thongsuriwong et al., 2010; Saidani et al., 2015; Zhu et al., 2016; Qin et al., 2016; Park et al., 2015). Aluminum Nitride (AlN), one of the III-IV compound semiconductors with a wurtzite crystal structure, is promising for a high piezoelectric performance due to its high thermal conductivity, wide energy band gap and high break down voltage for flexible electronics. In the light of the

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aforementioned, a flexible tactile sensor based on AlN doped ZnO thin films is investigated in this study.

With respect to ZnO thin films, many investigations had been reported. Anisimkim et al. in 1995 developed Li doped ZnO thin film, which was used to detect some combustible gases and methane. Bae et al. in 1999 fabricated ZnO and ZnO-CuO electrodes by sol-gel method and spin-coating method. In 2005, Beber et al. prepared ZnO thin films by spin coating on a glass substrate with subsequent sintering. They found that the resistivity of the films varied between 4600 Ωcm and 8500 Ωcm depending on the annealing conditions. The ZnO films revealed the strong (002)-oriented texture while the annealing temperature was above 450°C. Sagar et al. in 2007 prepared the sol-gel solution with MEA and fabricated ZnO thin film by spin coating. They observed that the PH values of ZnO solution was changed to a level of alkaline solution. A high-quality zinc oxide thin film possessed large grain size and low surface roughness was obtained with the increase of PH. Bhuvana et al. in 2011 analyzed the properties of AlN doped ZnO films grown by RF sputtering. They found that the incorporation of Al and N ions influenced the grain size of the co-doped films from the XRD studies. The efficiency of AlN doped ZnO layers for the optoelectronic devices can be enhanced by means of increasing grain size to achieve better optical and electrical properties. In 2014, Joshi et al. investigated the influence of post-deposition annealing on the transverse piezoelectric coefficient and the vibration sensing performance of ZnO thin films. They fabricated the ZnO films with different anneal temperatures (100°C to 500°C). Their results revealed that the ZnO film annealed at 300°C showed a relatively excellent result in the vibration sensing studies. Marana et al. in 2017 observed that the increase of pressure caused a more rigid structure with a major piezoelectric response along to the rigid direction of z axis. Consequently, ZnO can be applied in many electronic and piezoelectric devices, and can endure a high pressure environment without changing its electronic and structural properties. According to the abundant references, it could be realized that the characteristics of pure ZnO material are well revealed and its application is more extensive.

There are numerous methods to prepare AlN doped ZnO thin film, such as the sol-gel process, spray pyrolysis, pulse laser deposition, photo chemical deposition, molecular beam epitaxy, chemical vapor deposition and sputtering, etc. (Kakati et al., 2010). Many researchers had reported the preparations of the ZnO thin films by using the magnetron sputtering and CVD, however, both methods are expensive. Comparing to the other deposition techniques, the sol-gel technique has the advantages of the low cost and low substrate temperature process. In this study, the AlN doped ZnO crystalline phase of thin films is obtained and the crystallinity and surface morphology are analyzed by X-ray diffraction (XRD) and scanning electron microscope (SEM). Additionally, the force feedback measurement is also utilized to evaluate the piezoelectric properties of thin films. The purpose of this research is to fabricate a lead-free piezoelectric thin film, which could be used in the tactile sensor to assist the action adjustment. Actually, the piezoelectric film developed in this study can be further employed in numerous fields, such as the advanced applications of piezoelectric energy harvesters, surface acoustic wave (SAW) devices and et al. This study might also enhance the development of the fabrication techniques for the AlN doped ZnO piezoelectric films.

EXPERIMENTAL

First, the aluminum nitride doped zinc oxide precursor solution is synthesized by dissolving zinc acetate, aluminum nitride in the ethanol, ethylene glycol and triethanolamine (TEA) stabilizer. The molar ratio of ZnO remains at 1.0 M and the aluminum nitride is fixed at 0.2 M. The solution is rigorously stirred for 3 hours at 80°C, and waiting for the complete reaction for 1 day. Consequently, the aqueous solution of ZnO is revealed with high solubility, stability, and transparency. The material properties of the solution are shown in Table 1. Afterwards, this precursor solution of AlN doped ZnO is used to deposit the piezoelectric films for the fabrcation of tactile sensor.

Table 1 The material properties and the quantity of the ingredients for the AlN doped ZnO solution

Name	Chemical formula	Purity	Viscosity	PH
Zinc Acetate Dihydrate	$(Zn(CH_3COO)_2 \cdot 2H_2O)$	98 %	-	6-7
Aluminum Nitride	AlN	99.5 %, 65-75 nm, Hexagonal	-	7.5
Ethylene Glycol	$C_2H_6O_2$	99 %	21 CPS	6-7.5
Ethanol	C_2H_5OH	99.4 %	1.2 CPS	7
Triethanolamine	$C_6H_{15}NO_3$	95 %	15 CPS	10.5

In order to increase the hydrophilicity and improve the adhesion of the AlN doped ZnO solution on the polyimide substrate and copper substrate, the flexible polyimide substrate and copper substrate have to be pretreated properly by the atmospheric plasma. In the process of thin film deposition, the flexible polyimide substrate and copper substrate are at first cleaned by the atmospheric pressure plasma. Then, the AlN doped ZnO solution is deposited on the flexible polyimide substrate and copper substrate by the spin-coating method. The specimens are coated with different spinning speeds at 600, 800 and 1000 rpm for 18 seconds, and the number of the deposit films is 3 layers. Each as-coated film is baked at 150°C for 25 min to remove the organic solvent. The main reason of the baking process is to decrease the possibility of the scorch and crack happened in the thin films during the post-deposition annealing. Subsequently, the thin films are put into a high temperature furnace and processed with the annealing treatment at 250°C for 1 hour. During the annealing process, the lattice of thin films could be rearranged and the residual stress in the films would be eliminated. The parameters of the fabrication process are listed in Table 2. Furthermore, the flow chart of the preparation for the AlN doped ZnO thin films is illustrated in Fig. 1, and the schematic diagram of this tactile sensor is shown in Fig. 2. Each parameter of the AlN doped ZnO thin films is prepared and examined at least five times to confirm the reliability of the experiments. The crystalline quality of the thin films is evaluated by X-ray diffraction (XRD) performed by PANalytical X'Pert PRO MRD at 45 kV and 40 mA with Cu Kα radiation (λ=0.154 nm). The topography of the surface is observed by JEOL JSM-7600F, a high resolution scanning electron microscopy.

Table 2 The parameters of spin coating process

Spin coating	Heating	High temperature furnace
600 rpm	150°C for 25 min	250°C for 1 hour
800 rpm	150°C for 25 min	250°C for 1 hour
1000 rpm	150°C for 25 min	250°C for 1 hour

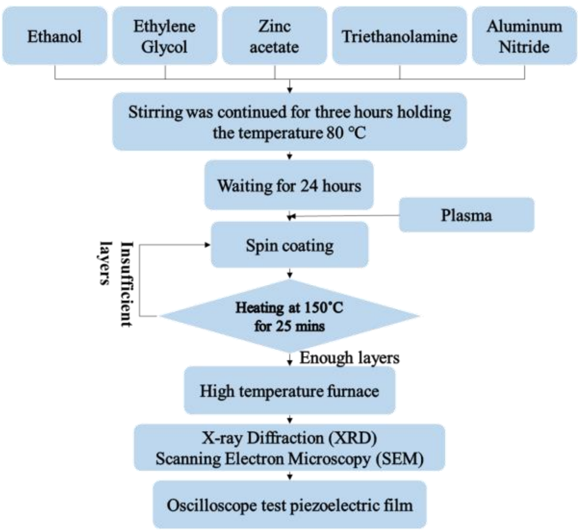

Fig. 1 The flow chart for the preparation of AlN doped ZnO thin films

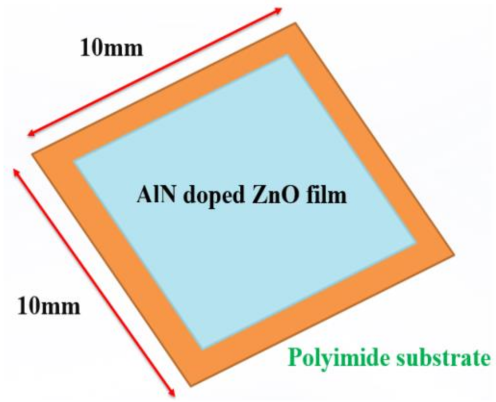

Fig. 2 The schematic diagram of tactile sensor based on AlN doped ZnO thin films

RESULTS & DISCUSSION

In this section, the influences of the parameters in the film preparation, such as the speed of spin-coating (600, 800, 1000 rpm) and different substrate (PI and copper), on the response of piezoelectric effect would be discussed. First, the phases of AlN doped ZnO films on the substrate need to be confirmed. Therefore, the X-ray diffraction is applied to investigate or identify the crystallinity of the thin film layers with different fabrication processes. The experimental results of XRD analysis for the thin films are shown in Figs. 3 and 4. In order to compare with these experiments conveniently, the JCPDs (Joint Committeeon Powder Diffraction Standard) card of ZnO (No. 01-079-0207) (JCPDS, 2015) is also demonstrated in Fig. 5.

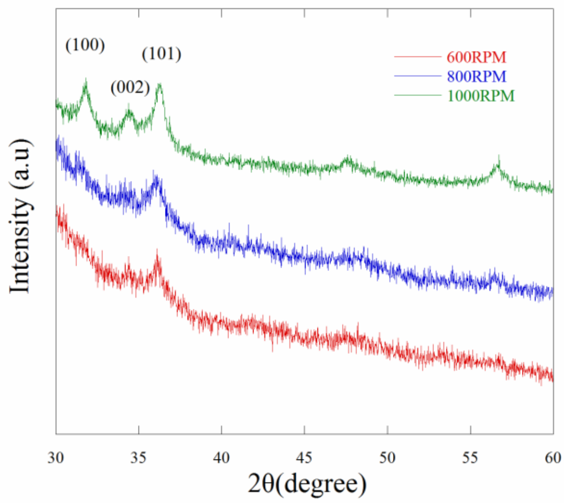

Fig. 3 The XRD of AlN doped ZnO thin films deposited on the PI substrate with different speeds of spin-coating (600, 800, 1000 rpm)

Fig. 3 shows the crystalline phases of the ZnO thin films with different speeds of spin-coating (600, 800, 1000 rpm) deposited on the PI substrate. In this figure, the crystalline planes of (100), (002) and (101) for ZnO structure could be indexed by JCPDs card. Accordingly, the result shows that the thin film layer entirely consists of (100), (002) and (101) oriented ZnO in all of the samples. As well known, the piezoelectric effect of the ZnO thin films crucially depends on the growth of the (002) lattice plane. Fig. 4 shows the crystalline phases the ZnO thin films deposited on the copper substrate under various speeds of spin-coating (600, 800, 1000 rpm). From this figure, it could be found that the diffraction peaks of ZnO thin films deposited on the copper substrate are higher and more distinct than those on the PI substrate. In both two figures, the highest peak appears at 1000 rpm, which might imply the best piezoelectric characteristics of these cases. For ZnO thin films, the structure is a hexagonal wurtzite, which consists of an oxygen and zinc atom held together by the ion bond. From our experiments, the intensity of X-ray diffraction increases gradually with the annealing temperature. Furthermore, the piezoelectric characteristics of the AlN-doped ZnO thin films are promoted with the increase of the speed of spin-coating.

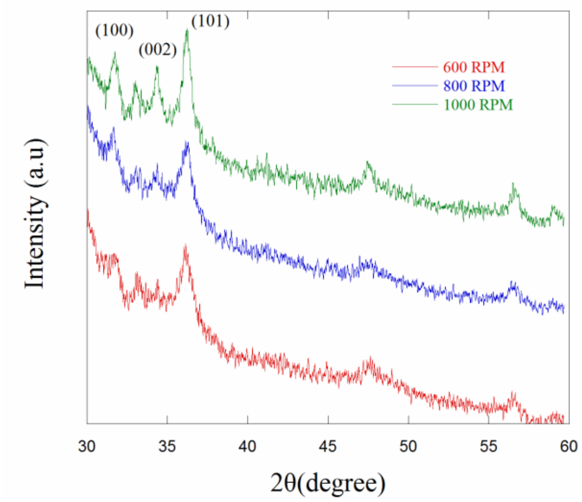

Fig. 4 The XRD of AlN doped ZnO thin film deposited on the copper substrate with different speeds of spin-coating (600, 800, 1000 rpm)

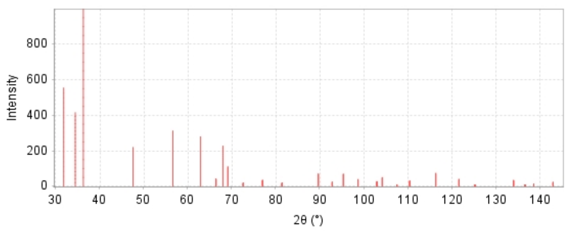

Fig. 5 The JCPDs card (No. 01-079-0207) of ZnO (JCPDS, 2015)

Next, the microstructures of the AlN doped ZnO thin films on the substrate are investigated. The topography of the film surface could be observed by SEM. The results of SEM observations with different speeds of spin-coating are presented in Figs. 6 (a)-(c). We observe that the refinement of the crystallinity in the AlN doped ZnO thin film is not clear. In these SEM images, it shows that the grains of ZnO films would form gradually as the speed of spin-coating is increased. This point of view is consistent well with the results of XRD analysis. It can be informed that the best speed of spin-coating is at 1000 rpm from XRD and SEM. Although, as the spin coating is getting faster, the better crystallization of the thin films might be gained. However, while the piezoelectric film becomes thinner due to the high speed of spin coating, it brings about the high resistance of the films and causes the reduction of the voltage. To reach a compromise, the higher speeds of spin coating are not chosen in the fabrication process. Consequently, the processing parameters with the speed of spin-coating at 1000 rpm are utilized to prepare the piezoelectric films in the experiments.

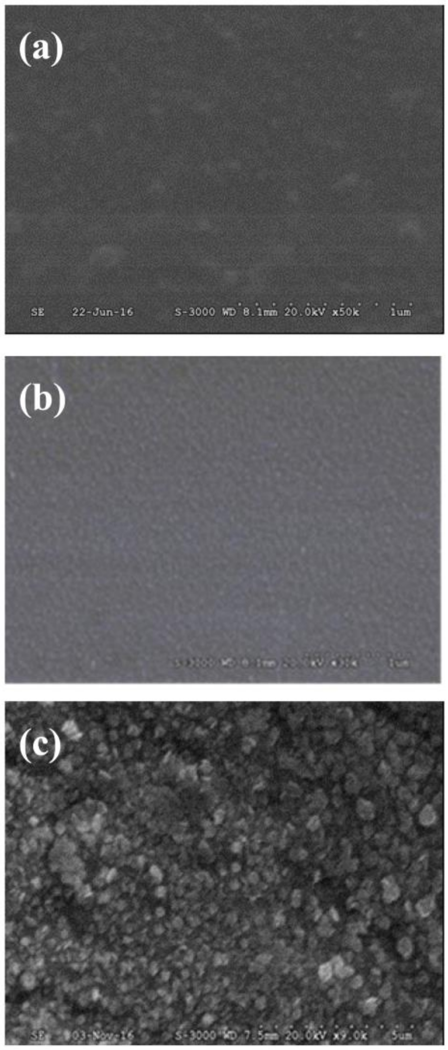

Fig. 6 The SEM photograph of AlN doped ZnO thin film, speed of spin-coating (a) 600 rpm (b) 800 rpm (c) 1000 rpm

Finally, the piezoelectric responses of the thin films are examined by the force feedback testing system. The apparatus of the force feedback testing system is shown in Fig. 7. The applied force is acquired from the driving vibrator of the dynamic testing platform.

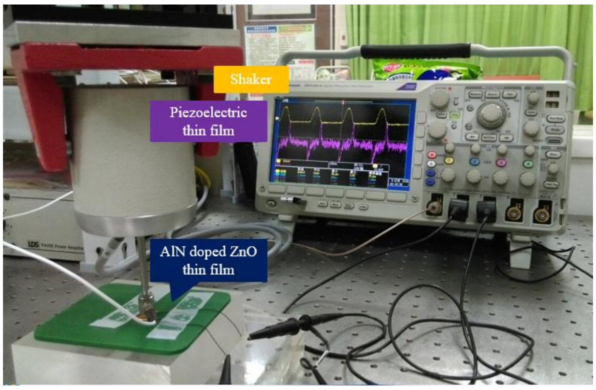

Fig. 7 The apparatus of the force feedback testing system

The output voltages of tactile sensors with different applied forces are demonstrated in this study. Fig. 8 shows the force feedback results of AlN doped ZnO films on the PI substrate with the spin coating at the speeds of 600, 800, 1000 rpm, respectively. The results in the figure are the averages of the five measurements for each sample. The output voltage of tactile sensor with the speed of spin-coating at 600 rpm deposited on the PI substrate is presented by the green line of Fig. 8. The output voltages are 5.7 mV and 17.5 mV, respectively, as the applied forces are 0.5 N and 2.5 N. The blue line in Fig. 8 exhibits the output voltage of tactile sensor with the speed of spin-coating at 800 rpm deposited on the PI substrate. As the applied forces are 0.5 N and 2.5 N, the output voltages are 7.32 mV and 23.2 mV, respectively. According to the red line in Fig. 8, the output voltage of tactile sensor with the speed of spin-coating at 1000 rpm deposited on the PI substrate is revealed. The output voltages are 12.6 mV and 37.2 mV, while the applied forces are 0.5 N and 2.5 N, respectively. It can be observed that the output voltage is proportional to the speed of spin-coating. In addition, it is found that the output voltage would increase linearly with the applied force. Hence, the response of this tactile sensor representing the piezoelectricity of this thin film on the PI substrate is rationally verified.

Fig. 9 shows the force feedback results of AlN doped ZnO on the copper substrate with the spin coating at the speeds of 600, 800, 1000 rpm, respectively. The output voltage of tactile sensor with the speed of spin-coating at 600 rpm deposited on the copper substrate is displayed by the green line in Fig. 9. The output voltages are 14.2 mV and 34.4 mV, respectively, while the applied forces are 0.5 N and 2.5 N. Based on the blue line of Fig. 9, the output voltage of tactile sensor with the speed of spin-coating at 800 rpm deposited on the copper substrate can be known. While the applied force is changed from 0.5 N to 2.5 N, the corresponding output voltages are 19.2 mV and 40.5 mV, respectively. In the red line of Fig. 9, the output voltage of tactile sensor with the speed of spin-coating at 1000 rpm deposited on the copper substrate is demonstrated. The output voltages are 23.5 mV and 54.3 mV, respectively, as the forces of 0.5 N and 2.5 N are loaded. Similar to the previous examinations, the output voltage is linearly raised with the increase of the applied force. The response of this tactile sensor induced by the piezoelectricity of this thin film on the copper substrate is distinctly identified from these measurements. Furthermore, it can be observed that the output voltage on the copper substrate is much higher than that on the PI substrate. One of the possible reasons may be attributed to the extraordinary capability of conducting electricity for a metal copper.

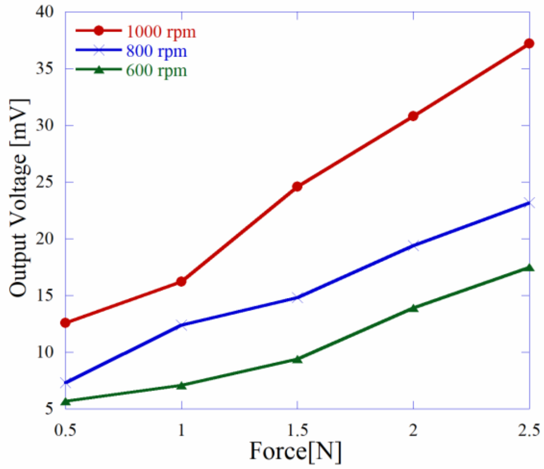

Fig. 8 Voltage response of AlN doped ZnO thin film tactile sensor on the PI substrate

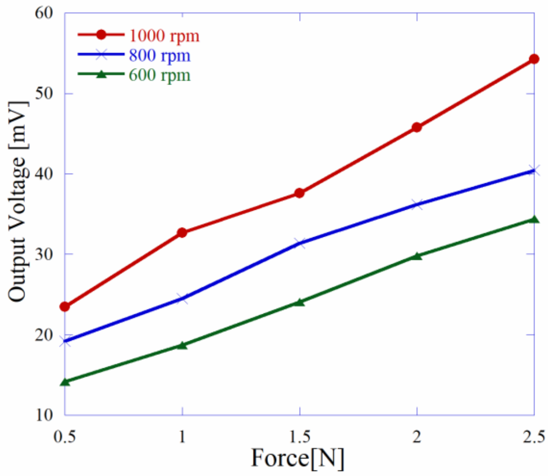

Fig. 9 Voltage response of AlN doped ZnO thin film tactile sensor on the copper substrate

CONCLUSION

In this study, the AlN doped ZnO solution is synthesized by the sol-gel method and the piezoelectric thin films are fabricated on PI substrate and copper substrate by the spin-coating method. We have investigated the influence of the speed of spin-coating with PI substrate and copper substrate on the crystallinity by XRD analysis. The effects of the speed of spin-coating on the topography of the surface are also demonstrated by SEM observations. In both of two experimental analysis, we have found that the speed of spin-coating at 1000 rpm is better than other speeds. We also perform the force-feedback analysis to compare the output voltages of ZnO films on PI substrate and copper substrate. It is observed the output voltage on the copper substrate is higher than that on PI substrate. This might be attributed to the better capability of conducting electricity for a metal copper. We have proved the availability and robust response of this tactile sensor according to the force feedback testing.

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參雜氮化鋁之氧化鋅壓電薄膜製備力回饋觸覺感測器研究

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摘要

本研究主要應用氮化鋁參雜之氧化鋅壓電薄膜來開發可撓式之觸覺感測器，此觸覺感測器可應用於複雜表面之元件。本觸覺感測器元件是利用溶膠凝膠法和旋轉塗佈法，將氮化鋁參雜之氧化鋅壓電薄膜製作於聚醯亞胺(PI)基板或銅基板上。本研究同時探討薄膜製程中旋轉塗佈轉速對於氧化鋅壓電薄膜結晶性及壓電效應之影響。透過X-ray繞射分析與掃描式電子顯微鏡來觀察所開發之氧化鋅壓電薄膜的結晶性質及表面形貌，並且對在不同基板上之壓電薄膜進行力回饋響應測試。由力回饋量測實驗中可證實本研究之可撓式觸覺感測器具有可行性與強化之壓電特性。