

## Study on Pizoelectric Thin Film for Micromachines(マイクロマシンのための圧電薄膜の研究)

著者	李 成 浩
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1] ソン ホ 成 名 浩 李 氏 学 付 博士(工学) 与 授 平成13年3月26日 学位授与年月日 学位授与の根拠法規 学位規則第4条第1項 研究科、専攻の名称 東北大学大学院工学研究科(博士課程)機械電子工学専攻 学位論文題目 Study on Piezoelectric Thin Film for Micromachines (マイクロマシンのための圧電薄膜に関する研究) 正喜 遵 教 官 東北大学教授 江刺 指 論文審查委員 東北大学教授 江刺 正喜 主査 東北大学教授 羽根 一博 東北大学教授 新妻 弘明

## 論 文 内 容 要 旨

This thesis describes the deposition method and microsystems applications of piezoelectric thin film, Pb(Zr<sub>1-x</sub>Ti<sub>x</sub>)O<sub>3</sub> material. There has been research with piezoelectric thin film angular rate sensor, however the good properties of PZT thin film for microsystems or MEMS application were not obtained yet. For microsystems applications of piezoelectric PZT thin film material, the PZT material must have more than thickness of 1  $\mu$  m. The development of deposition more than thickness of 1  $\mu$  m for microsystems or MEMS applications is still under working. In our research, we had worked the PZT thin film of more than thickness of 1  $\mu$  m using sol-gel method and evaluated the angular rate sensor using this material.

Chapter 1 appropriately introduces the motives of the research. Piezoelectric actuation using bulk ceramic materials is well known, but widespread use in MEMS requires suitable deposition and integration methods, which are compatible with large-scale manufacturing. One primary factor limiting the development of piezoelectric MEMS has been the lack of a suitable thin film piezoelectric material, PZT and its derivatives have excellent piezoelectric properties and therefore are logical choice for MEMS applications. Piezoelectric materials are one type of the functional materials utilized in MEMS technology.

Chapter 2 presents the theory and evaluation of the piezoelectric material. Pb (Zr, Ti)O<sub>3</sub> is complete solid solution of PbZrO<sub>3</sub> and PbTiO<sub>3</sub> the morphotrophic phase boundary (MPB), which

shows the maximum dielectric and piezoelectric property at the point. In order to be used  $a_8$  actuators and sensors, the PZT composition must become the morphotrophic phase boundary.

Piezoelectricity is an electromechanical phenomenon coupling the electric and elastic fields in piezoelectric material. The 32-point groups are subdivisions of seven basic crystal systems, which are, in order of ascending symmetry, triclinic, monoclinic, orthorhombic, tetragonal, rhombohedral, hexagonal, and cubic. Of the 32-point groups, 21 classes do not posses a center of symmetry (a necessary condition for piezoelectricity to exist) and 20 of these are piezoelectric. The perovskite crystal structure of PZT material belongs to 4mm point group. When a piezoelectric material is stressed, and electric field is generated in the stressed region; on the other hand, if an electric field is applied, there will be an induced stress on the material in the region of the field. These effects are related with the applications of sensor and actuator of the piezoelectric materials.

Chapter 3 discusses the deposition methods of piezoelectric thin film and shows the results of the piezoelectric thin film using sol-gel method. The sol-gel method offers a unique combination of precise control of chemical composition easily, uniformity over large areas, relatively low process temperatures that are compatible with integrated silicon processing and low cost. The sol-gel solutions were made by alkoxide method. A precursor solution of sol-gel PZT is composed of trihydrated lead acetate, titanium iso-propoxide, zirconium iso-propoxide and 2-methoxyethanol for solvent. The composition of the precursor solution for 0.5M Pb( $Zr_{0.53}$  Ti<sub>0.47</sub>)O<sub>3</sub>, which is corresponded to morphoropic phase boundary(MPB). The PZT thin film was coated on the Pt/Ti/SiO<sub>2</sub>/Si structure. The crystal structure of PZT film by rapid thermal annealing showed the pervoskite phase with (100) crystal orientation. Thickness of  $1.5\,\mu$  m was obtained without cracking. The process consists of 15-times repeat of the multiple spin-coating and RTA of 650°C for 1 min. The PZT thin film showed the remnant polarization of  $9.9\,\mu$  C/cm² and maximum polarization of  $54.4\,\mu$  C/cm² respectively. The electrochemical constants of PZT thin film showed the value of susceptance(B) of  $4800\,\mu$  s at capacitance of 790pF.

Chapter 4 explains the microfabrication for PZT thin film devices. The PZT thin film was etched by wet etching method. In order to etch ferroelectric thin film like Pb(Zr, Ti)  $O_3$  (PZT), a wet etch process is commonly used. PZT material is etched in HCl or HNO<sub>3</sub> solution with some drops of HF usually. The used etching solutions are HCl/H<sub>2</sub>O/HF = 30/70/5, HNO<sub>3</sub>/H<sub>2</sub>O/HF = 5/4000/1. But, the etching was critical conditions because the etching rate and the underetch are

strongly dependent on the etching solution and the PZT density. We etched the sol-gel PZT thin film using the ICP RIE. The masking material was Cr thin film with thickness of  $0.6\,\mu$  m. The etching was performed at the conditions of r.f. stage power of 130 watt and coil power of 150 watt for 18min in the vacuum pressure of 3mtorr. The SF<sub>6</sub> gas was used as the etching gas. The layers from PZT thin film to silicon wafer could be etched by this ICP RIE method simultaneously.

Chapter 5 focuses the applications of the PZT thin film for Microsystems. First, the PZT thin film cantilever structure was fabricated. The lower electrode, Pt, was made by lift off method. The PZT thin film was etched by wet etchant. The PZT thin film cantilever beam was released using deep reactive ion etching method. The method for calculating the piezoelectric coefficient (d<sub>31</sub>) was suggested with mathematical equations. The PZT thin film angular rate sensor for Microsystems was considered. The PZT thin film angular rate sensor was designed in order to drive and sense simultaneously. The basic principle of piezoelectric devices is that piezoelectric materials induce a charge or develops a voltage across itself when it is deformed by stress. The output values from the sensor are amplified in a charge amplifier, which converts the charge generated by transducer sensor into a voltage that is proportional to the charge.

The fabrication of PZT thin film angular rate sensor was performed by bulk micromaching method. From the fabricated PZT thin film angular rate sensor, we obtained the driving amplitude of  $0.31\,\mu$  m by the vibration the PZT thin film with 2Vp-p driving voltage of 180 phase difference at resonant frequency of 35.8kHz. The oscillating output voltage obtained by external actuation using a stacked piezoelectric actuator showed the values of 0.76V and 0.87V in outer inner driving electrode at driving voltage of 5Vp-p.

PZT thin film angular rate sensor could be oscillated by PZT thin film. However the output of the angular rate signal was not obtained. The reason is that PZT thin film angular rate sensor had different displacements and resonant frequencies with positions.

Chapter 6 suggests for future research. The accurate design of piezoelectric materials is needed to know the accurate behavior of electrically induced-strain distribution. And for micromachines application using piezoelectric thin film, it will be necessary to study the conditions of wet etching and dry etching

## 論文審査結果の要旨

シリコンのマイクロマシニング技術を用いるといろいろな要素を小形に集積化したマイクロマシンを実現することができ、この技術は情報機器周辺の鍵をにぎる重要な部品を製作するための共通基盤となっている。しかしアクチュエータと呼ばれる運動要素に高性能なものが望まれており、これには圧電材料などの機能性材料の技術とシリコンのマイクロマシニング技術を組合わせた技術が必要である。

本論文は、圧電材料である PZT(チタン酸ジルコン酸鉛)を薄膜としてシリコン構造体の上に形成し、それを振動ジャイロに応用する研究を行ったもので、全編6章よりなる。

第1章は緒論である。

第2章では、圧電材料の理論や評価法についてまとめている。

第3章では、圧電材料薄膜(圧電薄膜)の堆積法とその特性に関して述べている。ゾルーゲル法を用いて堆積しているが、その後の急速熱処理の条件などが重要である。またアクチュエータとして使うには  $1 \mu$  m以上の厚さが要求されるため、これらの工程を繰り返す必要がある。この研究で比較的厚い圧電薄膜を割れずに形成できたことは貴重な結果である。

第4章では、デバイスの作製に関する技術について述べている。PZT 薄膜のエッチングに液体を用いる方法と気体中で行う方法を比較し検討している。

第5章では、圧電薄膜をマイクロマシンへ応用した結果について述べている。シリコンの片持ち梁に形成して、その振動特性を評価した。またシリコン振動ジャイロに形成して、振動駆動や振動検出などを行っている。

第6章は結論である。

以上要するに本論文は、マイクロマシンに適合する圧電薄膜について研究を行ったもので、マイクロマシン工学ならびに機械電子工学に寄与するところが少なくない。

よって、本論文は博士(工学)の学位論文として合格と認める。