Fabrication and characterization of PDC SiCN RTD sensor

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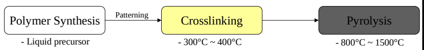
Abstract

In this paper, we describe the fabrication of ceramic thin films for high-temperature heat flux sensors. The silicon carbon nitride (SiCN) thin films are prepared by using soft lithography on preceramic polymer precursors followed by pyrolysis and heat treatment. Processing routes have been developed which lead to thin film Resistance-Thermal Detectors (RTD) that have sufficient mechanical strength for handling and for use in thermal sensing. The annealing temperature can be used to control the conductivity of the resulting sensors. The electrical resistivity of the sensors was measured at different temperatures from 1200-1400°C.

Introduction

❖ What is Polymer Derived Ceramics(PDC)?
- Introduced in the late 1960's by chantrell et al.
- Conversion from preceramic polymer to ceramic by heat
- Fabrication process



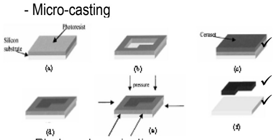
❖ Advantages over conventional ceramics
- More complex and micro/nano scale shapes possible by MEMS techniques
- Less pyrolysis temperature(800°C ~1500°C)
  ➔ inexpensive, easier process
- Able to tailor the properties of ceramics by controlling fabrication conditions
  ➔ Suitable for high temperature MEMS

❖ Why Polymer Drived SiCN
- Superior strength and thermal shock to other ceramcis
- MEMS techniques(lithography, etc.) are applicable : ➔ 3D structures possible
- Inexpensive than conventional ceramics

➔ Noticed as a substitute material for Si wafer for high temperature application

❖ Applications

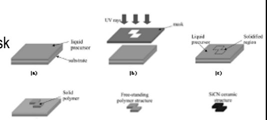
❖ Two major fabrication method for PDC SiCN
- Micro-casting



✓ micro molding by photoresist
✓ precursor spun on mold
✓ crosslinking and pyrolysis

- Photo-polymerization

✓ precursor spincoating
✓ UV exposure through mask
✓ rinsing with acetone
✓ free standing structure and heat-treatment



Objectives

❖ To contribute to current flow of PDC SiCN MEMS in terms of fabrication process
- By obtaining a free standing SiCN with new approach in polymerization method

※Direct contact polymerization is introduced

❖ To investigate the characterization of our own PDC SiCN for better use as a RTD
- Composition analysis by SEM/EDS over the cross-section of SiCN film
- Composition change after oxidation and Crystallization test by XRD
- To get the data on temperature dependency of electrical conductivity of SiCN

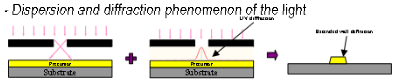
➔ To make a better PDC SiCN RTD sensor

Fabrication of PDC SiCN

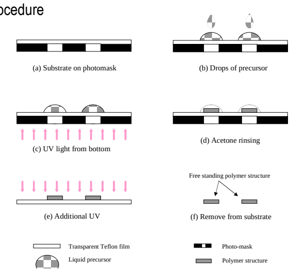
❖ Problems in photo-polymerization
- Waste of polymer precursor ; caused by spincoating whole wafer to get targeted thickness of precursor

❖ Difficulties in setting to make the gap as narrow as possible : attachment of photo-mask and precursor

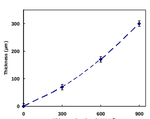
❖ Extended wall definition ; caused by the gap between mask and precursor
- *Dispersion and diffraction phenomenon of the light*



❖ Novel direct contact polymerization technique
- procedure

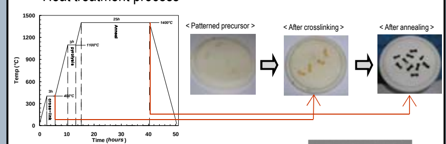


❖ Advantag
- No need to spincoat the precursor
  ➔ expense and time saving
- The thickness can be controlled by UV exposing time
- Easy removal from the substrate : Teflon

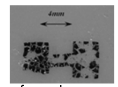


❖ Heat treatment
- Important process : properties decided
- Performed in high temperature Tube furnace with flowing purified N2 gas
- Consists of three steps : 1) Cross-linking, 2) Pyrolysis, 3) Annealing at 400°C, 1100°C, 1400°C respectively

- Heat treatment process



- Some failures during heat treatment
  ► Due to thermal stress, samples break into the pieces
  ► To avoid failures, 1) complete solidification of sample 2) aged patterns for 12 hours at room temperature

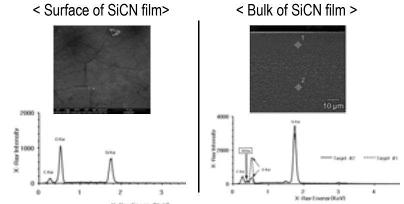


- Successful PDC SiCNs

Characterization of PDC SiCN

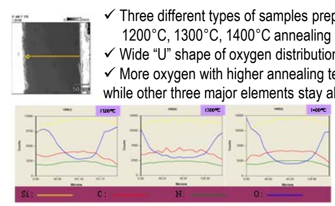
❖ Composition analysis by SEM/EDS

< Surface of SiCN film> < Bulk of SiCN film >

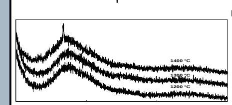


- Surface has more oxygen, likely in the form of oxide layer
- In the interior(target #2), the amount of oxygen reduced

❖ Depth dependence of composition of SiCN
- Linescaning analysis conducted over about 200μm thick samples
  ✓ Three different types of samples prepared with 1200°C, 1300°C, 1400°C annealing temperature
  ✓ Wide "U" shape of oxygen distribution for samples
  ✓ More oxygen with higher annealing temperature while other three major elements stay almost same

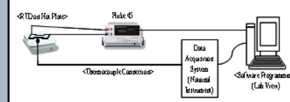


❖ Crystallinity test
- X-Ray Diffraction(XRD) was performed on SiCN samples
  ► Three samples are annealed at 1200°C, 1300°C, 1400°C



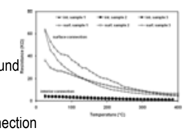
► Results
- For samples of 1200°C, 1300°C, no sharp diffraction peaks appear
- For samples of 1400°C, small diffraction peak appear
  ➔suggesting the onset of crystallization

❖ Electric resistance measurement
- Configuration for data acqusition

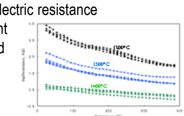


< Features >
✓ resistance measured by Fluke
✓ temperature measured by thermocople
✓ both data recorded in Lab view program

❖ Effect of increased oxygen content near surface
- Electric connection made in two ways : surface and interior connection
- Measurement Results
  1) Resistance of the sample with ground surface was much lower
  2) More wide range of resistance at room temperature for surface connection
  3) Ground samples has less variability of resistance



❖ Effect of annealing temperature on electric resistance
- 4 SiCNs samples with three different annealing temperature are prepared
  ► Selected samples are surface ground for interior connection
- Measurement results
  ► magnitude of resistance depends on the annealing temperature
  ► Resistance of SiCN RTD annealed at 1200°C is larger than 1400°C by the 2 orders of magnitude



Conclusion

◆ We have developed a novel photo-lithography method, which is Direct Contact Lithography.
- Without the gap between mask and precursor, better dimensional control of the developed pattern is possible
- Exposure time was used to control the pattern thickness
  ➔ we can obtain desired thickness without using spincoating
- Teflon think film as a substrate and additional UV exposure facilitates the removal of pattern from the substrate

◆ Characterization by XRD was conducted for our fabricated PDC SiCN.
- At low annealing temperatures, the SiCNs are amorphous but some crystallinity appears at the higher annealing temperatures

◆ SEM/EDS results show that our SiCNs have a surface oxide layer and this surface layer has a significant effect on the electric conductivity of the sensors

◆ By electric resistance measurement of SiCN,
- The resistance can be controlled by annealing temperature
- The temperature dependence of the electric resistance shows that the RTDs can be used for temperature sensing, which can be further used for heat flux measurements in high temperature environment

References

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Acknowledgement

◆ This research is funded by US Army Program Executive Office