Design and Fabrication of a Micro-size Thermionic Ionization/Flame Ionization Detector for Gas Phase Chemical Analytes

Polysilicon air-bridge filament heater with integrated electrodes

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Motivation

- Portable chemical detection becoming more necessary in today’s world
- Need a sensitive device that can detect a large variety of chemical species
- The detector needs to fast
- To design a device in a way that can be fabricated and packaged easily

Micro-sized TID/FID
Ionization Detector and GC analysis

Gas Chromatography
- Method for separating mixtures into individual components
- Very reliable method
- Has matured over a half a century

Why a Ionization detector?
- Sensitive to a larger variety of organic and phosphorus and nitrogen containing compounds
- PPB detectability
- Have “control” over the sensitivity
- Very fast
- Can be made very small

Sample output from a GC with 22 peeks (Ref: [1])
Theory of Ionization Detection

- Polysilicon Micro-filament heater

- Make hot
- Thermionic emission occurs causing ionization
- Force ions to a collection plate
- Measure resulting current or voltage
Modes of Operation

Thermionic Ionization Detector
- Can detect many different species if hot enough
- Requires more power than other detectors

Nitrogen-Phosphorus Detector
- Coat heater with Alkali salt
- Lowers work function
- Sensitivity increase for nitrogen and phosphorus containing compounds

Flame Ionization Detector
- Coat heater with platinum base compound
- Add H₂ fuel
- Catalytic combustion occurs, ionizing species (chemi-ionization)
MEMS surface micromachining techniques utilized

Simple, three mask level process

1. Deposit 5000Å silicon nitride

2. Deposit sacrificial oxide, 3µm PECVD TEOS

3. Pattern and etch sacrificial oxide in BOE

4. RCA clean and deposit 2µm of polysilicon
5. Coat poly with N-type spin-on-glass

6. Drive dopant in at 1050°C for 120 minutes, Strip off SOG in BOE

7. Deposit 7500Å aluminum via Sputtering

8. Pattern and etch Al and polysilicon to isolate structure (RIE)

9. Pattern and etch Al (wet)

10. Remove sacrificial oxide in 49% HF
Micro-heater Designs

- Straight, Serpentine and Mesh heater designs
- Electrodes places 100 to 500µm from heating elements
- Designed to be low resistance (100Ω)
- Heat to 800-900°C with 15 to 20V
- Die dimensions: 4.5 x 6.5mm
**Fabrication Issues**

- **Slow sacox etch**
  - About 4100 Å/min (Literature says about 18,000 Å/min in 49% HF)
  - Possible carbon build up on sidewalls during poly etch
  - Clean in O₂ plasma increased etch rate to about 7500 Å/min

- **Capillary forces prevent complete structure release**

  Vapor bubbles forming under poly structure when heated

  *Rectified by rinsing after release in DI Water / IPA / DI Wafer / IPA and baking at 200°C for 10 minutes on a hotplate*
Testing of TID: TCR and Heating

- Resistance of heater dependant on both on thermal coefficient of expansion and thermal coefficient of resistance

- At higher temperature, more intrinsic carries form but also the size of the heater resistor gets larger and deflects upwards

\[
\left( \frac{R}{R_0} - 1 \right) \frac{T}{T - T_0} = TCR
\]

- Average TCR \(\sim 0.000209/°C\)

Estimated Temp: 877.8 °C
46V, 68mA R = 705Ω

3mm x 300µm Heater filament
Testing of TID: Chemical Detection

**Current Setup**

**Diagram:**
- Nitrogen
- 1/4in Tubing
- Swage Union Tee
- Cotton Swab Soaked in Acetone
- Testing Probes
- TID Chip
- Chuck

**Test Package Fabrication**
Modified TO-8 IC can

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Testing of TID: Chemical Detection

- Used HP4145A as a glorified picoammeter
- 100V Bias applied to plate

Chip 1 Signal 5/9/2004 8:18AM
Analyte: Acetone, 100V Bias, Heater already on 50V, 80mA

Current (mA)

Time (sec)

Acetone Present

Detector Response
Testing of TID: Chemical Detection

- Signal not as clean as first run, but present
- Turning on heater may ionize surface contamination

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Conclusion

- Successful fabrication of a micro-heater filament with integrated electrodes, using doped polysilicon was performed using MEMS surface micromachining techniques.

- By measuring the TCR of the micro-heaters, it was estimated that temperatures above 800°C were attained.

- Successful detection of a gaseous chemical analyte, acetone, was performed.
Future Work

- To quantify the lower detectability limit of TID
- Redesign of the device to operate at low power and higher temperature
- Test the device in both the NPD mode and FID mode
- Research lower work function materials, that are “fab friendly”, for heater designs
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Test Package Fabrication

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References


