A LIGA Fabricated Two-Dimensional Quadrupole Array for High Resolution Mass Spectroscopy

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Project Objectives and Approach:

The objective of this project is to build a functional miniaturized Quadrupole GC/MS system by advancing the state-of-the-art of key components which in turn will be integrated with other elements currently under development as NASA SBIR projects. This project seeks to leverage previous investments in NASA funded advanced technology projects to realize the functional components of the system.
LIGA is a German acronym derived from Lithographie, Galvanoformung and Abformung which are interpreted as lithography, electroplating and replication. It is an X-ray lithography technique in which a polymer film is exposed to x-ray radiation which breaks some of the polymeric bonds causing a reduction in molecular weight of the exposed areas. This difference in molecular weight is then exploited to dissolve away the exposed areas leaving a mold into which metals are electroplated. This can be used as a final part or is the basis for further replication using techniques such as injection molding.
Advantage of LIGA micromachining

- Able to produce complex microstructures
- High Precision (good dimension control)
  - Very small structures (10 micron range)
  - High Aspectio ratio (upto 100)
  - Vertical Sidewalls (1 micron / 1000 micron slope)
  - Very smooth sidewalls (variation less than 30 nm)
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GC/MS System Block Diagram

- Sample Path
- Gas Chromatograph
- High Vacuum Region (approx. 10-6 Torr)
- Mass Filter
- Vacuum Roughing Pump
- High Vacuum Pump

Symbols:
- □ = NASA SBIR Projects
- □ = To be developed under this project
- □ = COTS, or technology integrated from other efforts

Power Source

- High Precision, 50 Mhz RF Power Supply
LIGA is a German acronym derived from Lithographie, Galvanoformung and Abformung which are interpreted as lithography, electroplating and replication. It is an X-ray lithography technique in which a polymer film is exposed to x-ray radiation which breaks some of the polymeric bonds causing a reduction in molecular weight of the exposed areas. This difference in molecular weight is then exploited to dissolve away the exposed areas leaving a mold into which metals are electroplated. This can be used as a final part or is the basis for further replication using techniques such as injection molding.
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Structure Length (micron)

1,000  2,000  3,000

Energy Density of Synchrotron X-ray Source

Center for Advanced Microstructures and Device (CAMD)

Sandia National Lab

Brockhaven National Lab

JPL

CALIFORNIA INSTITUTE OF TECHNOLOGY

NASA
Fabrication Steps

- Preparation of X-ray mask
  - Thick photoresist patterning on silicon wafer
  - Electrodeposited Gold mask
- PMMA bonding
- Synchrotron X-ray radiation of PMMA
  - Break down of the long molecular chain (750,000 g/mol) to shorter chain (3000 to 6000 g/mol)
- X-Ray Resist Development
- Electroplating
- Dissolving the PMMA mold
- Planarization
Thick Film Lithography

To make the thick absorber X-ray masks necessary to operate at the more energetic synchrotron sources such as the Stanford Synchrotron Radiation Lab (SSRL) at Stanford Linear Accelerator Center and the National Synchrotron Light Source (NSLS) located at Brookhaven National Lab, techniques in thick film UV lithography have been developed. These techniques can be used to generate LIGA like structures. Although patterns greater than 1 mm can be generated, the aspect ratio, wall angle and wall straightness of the full LIGA process can not be matched using these methods. The thick film lithography techniques have proved adequate for several devices with nominal thickness' under 100 microns.
Electroplated Gold Mask
(X-ray absorber)
X-rays used in the LIGA process are obtained from a synchrotron source such as the Advanced Light Source (ALS) located at Lawrence Berkeley National Lab. In these devices electrons are accelerated to relativistic speeds and held in a storage ring consisting of straight and curved sections. As the path of the electrons is bent, energy is released in the form of X-ray radiation.
Synchrotron X-ray exposure

3 and 4” Wafer Holder

Scanner

Synchrotron Source

Distribution [a.u.]

Molecular weight [kg/mol]
Developing
Electroplating

Electroplating Stations with fine temperature and process control

Electroplating Paddle Cell
Electroplating

Developing electroplating solution to enhance mass transfer in deep trench and promote uniform growth (eliminate dendrite and powdery growth)

Developing electroplating solution and processes to minimize internal stress in the deposits
Electroplating apparatus

Cathodic (Reduction) Reaction

e.g.
Ni^{+2} + 2e^- → Ni_{(s)}
2H^+ + 2e^- → H_2 (side reaction)

Anodic (Oxidation) Reaction

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Ni_{(s)} → Ni^{+2} + 2e^-
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Developing electroplating solution and processes to minimize internal stress in the deposits
Stress in Electroplated Cu

SEM micrographs of electroplated Cu

- 5 mA cm$^2$
- 10 mA cm$^2$
- 20 mA cm$^2$
- 40 mA cm$^2$

Graphs showing:
- Stress (MPa) vs. Current Density (mA/cm$^2$)
- Growth Rate (micron/min) vs. Current Density (mA/cm$^2$)
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Stress in Electroplated Ni

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Current Density (mA/cm²)

2.5 mA cm⁻²
10 mA cm⁻²
20 mA cm⁻²
40 mA cm⁻²

Growth Rate (micron/min)

Current Density (mA/cm²)
JPL Fabricated Miniaturized Quadrupole Mass Filters

- Conventional Machining
- Approximately 25 mm pole length
- 16 poles, 9 Quadrupoles
- 2 dimensional array
  (Chutjian, Orient, et al)

- Electro-Discharge Machining (EDM)
- Approximately 7 mm pole length
- 4 poles, 1 Quadrupole
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- LIGA Micromachining
- Approximately 3 mm pole length
- 20 poles, 9 Quadrupoles
- Linear array
  (Wiberg, Chutjian, Orient, et al)
LIGA Micromachined 2-D Quadrupole Arrays

Material:
Non-magnetic Copper
Pole Length: 3 mm pole
# of poles : 24 poles
# of quadrupole: 9 Quadrupole

3mm thick NiTi microhold
LIGA Fabricated 3 X 3 Arrays in 3" wafer
LIGA Fabricated Linear Quadrupole Array

Quadrupole Mass Filter

- Entrance aperture
- Dielectric spacer
- Quadrupole array
- Dielectric plating base
- Exit aperture
Relative Precision Requirements for Quadrupole Fabrication

Precision Machining Application Domain

- City: 10 km
- House: 1 km
- Arm: 1 m
- Optical fiber: 100 µm
- Bacteria: 1 µm
- Virus: 0.1 µm
- Atom: 1 Å

Relative Tolerance

- 100% 10% 1% 0.1% 0.01% 0.001% 0.0001% 0.00001%

Region of interest for microscale quadrupole fabrication

Quadrupole diagonal measure and percent deviation from mean (Ni Quadrupole 1-II)

- Diagonal measure (mm)
- % Deviation from mean

Linear dimension
Acknowledgements

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Developing

![Graph showing solvability vs. molecular weight][1]

[1]: Graph showing two curves indicating solvability percentages over molecular weight ranges. One curve is labeled as 'new' and the other as 'GC'.
Electroplating

Electroplating Stations with fine temperature and process control

Electroplating Paddle Cell
Electroplating apparatus

Cathodic (Reduction) Reaction

e.g.
Ni^{+2} + 2e^- \rightarrow \text{Ni}_{(s)}
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Anodic (Oxidation) Reaction

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\text{Ni}_{(s)} \rightarrow \text{Ni}^{+2} + 2e^-

- Anode
- Substrate
- PMMA (mold)
- Substrate holder
Electroplating

- Developing new electroplating solution to enhance mass transfer in deep trench and promote uniform growth (eliminate dendrite and powdery growth)
- Developing electroplating solution and processes to minimize internal stress in the deposits
Stress in Electroplated Cu

Current Density (mA/cm²)

Stress (MPa)

Growth Rate (micron/min)

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5 mA cm²

10 mA cm²

20 mA cm²

40 mA cm²
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Material:
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Pole Length: 3 mm pole
# of poles : 24 poles
# of quadrupole: 9 Quadrupole

3mm thick metal foil

Diagram showing the arrangement of the quadrupole arrays.
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Quadrupole Mass Filter

Entrance aperture
dielectric spacer

Quadrupole array

Exit aperture
dielectric plating base

Hyperbolic pole face array of 20 poles forming a linear array of 9 quadrupoles. Pole lengths are 3.3 mm.
Relative Precision Requirements for Quadrupole Fabrication

Region of interest for microscale quadrupole fabrication

Precision Machining Application Domain

Fundamentals of Microfabrication
MARC MARCOU
CRC PRESS
1997
## Acknowledgements

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The 3rd Workshop on Harsh-Environment Mass Spectrometry

and the 2nd NASA/JPL Pumps Workshop

March 25-28, 2002
Pasadena, CA
LIGA Fabricated Linear Quadrupole Array

Quadrupole Mass Filter

- Entrance aperture
- Dielectric spacer
- Quadrupole array
- Dielectric plating base
- Exit aperture

Hyperbolic pole face array of 20 poles forming a linear array of 9 quadrupoles. Pole lengths are 3.3 mm.
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The 3rd Workshop on Harsh-Environment Mass Spectrometry

and the 2nd NASA/JPL Miniature Mass Spectrometer Pumps Workshop

March 25-28, 2002
Pasadena, CA
Purpose: In situ mass spectrometry (MS) in a wide variety of harsh environments—from outer space to Earth’s oceans to battlefield scenarios—is rapidly becoming a reality. There are many common features to MS deployment in these vastly different conditions, including high reliability, small size, and low power requirements. The Harsh-Environment Mass Spectrometry (HEMS) Workshop will encourage interaction among those working on deployment of mass spectrometers in various harsh environments. The Miniature Pumps Workshop is inspired by the surge in development of miniaturized vacuum-dependent instrumentation such as mass spectrometers, charged particle analyzers, electron columns, and sublimation cells, to cite a few examples.

Technical Program: Talks/posters for the HEMS Technical Sessions will be selected for their focus on making mass spectrometer components and systems rugged and portable; interfacing mass spectrometers to the environment; autonomous sampling strategies; unattended operations; adaptive sampling; data processing and communications; enabling technologies; and miniaturization. The focus for the Miniature Pumps Workshop will be applications requirements, state-of-the-art pumping technology, the fundamental operating characteristics of different approaches, and technological limits to performance with decreasing size and mass. Rough and high-vacuum technologies will be covered, with an emphasis on miniaturization. Each session (except Session IV) will begin with an invited speaker.

Abstracts: Those interested in presenting a poster or talk should submit an abstract (maximum 500 words, in English). Submission deadline: December 15, 2001. Submission instructions are available on the Workshop website. The Proceedings will be distributed at the workshop. Please contact Ellie Trevarthen if you cannot access the website.

General Information: The workshop will be held at the Courtyard Los Angeles Old Pasadena, 180 North Fair Oaks Avenue, Pasadena, CA 91103, Ph: (626) 403-7600, Fax: (626) 403-7700. It is 40 miles from the Los Angeles Int’l. Airport, 12 miles from the Burbank Airport. The Courtyard Old Pasadena is in the heart of the revitalized “Old Town” Pasadena Historic District; a short walk from your room you will find many shopping, dining, and entertainment choices. More information and directions can be found at http://www.courtyard.com/.

Registration: $150 for two-day HEMS Workshop, $75 for one-day Miniature Pumps Workshop, $225 for both Workshops. Download the registration form from the conference website. Payment: by credit card or bank draft (in U.S. Dollars, drawn on a U.S. bank). The fee includes Workshop costs, Proceedings, continental breakfast and lunch each day, and Wednesday evening dinner (transportation and accommodations not included). Registration deadline: February 1, 2002. Refund for cancellation: request must be received by February 1, 2002 ($50 processing fee will be deducted). Registration is limited to 100 participants.

Accommodations: A block of rooms has been reserved at the conference hotel at the rate of $99 per night. Please contact the hotel directly to make your reservation; mention “HEMS 2002” to receive the conference rate. The hotel may fill up quickly, so we encourage you to make your reservation early. For other hotels in the area, contact Ellie Trevarthen.

Sponsors: Corporations interested in participating in the vendor exhibit should complete the Corporate Sponsor Form at the conference website and submit it to Ellie Trevarthen by December 15, 2001. Contact Ellie Trevarthen for assistance, if needed.

WORKSHOP CONTACTS
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TENTATIVE AGENDA

Monday, March 25
7:00 P.M. WELCOME RECEPTION AT COURTYARD OLD PASADENA

Tuesday, March 26
8:00 A.M. CONTINENTAL BREAKFAST
8:30 A.M. TECHNICAL SESSION I: SPACE ENVIRONMENTS
   INVITED SPEAKER: Jack Beauchamp, California Institute of Technology
   TOPIC: “NOVEL MASS SPECTROMETRIC APPROACHES TO THE IN SITU CHEMICAL
   ANALYSIS OF GALACTIC AND COMETARY DUST PARTICLES”
12:00 NOON INFORMAL LUNCH BUFFET
1:30 P.M. TECHNICAL SESSION II: MASS SPECTROMETERS FOR UNDERWATER APPLICATIONS
   INVITED SPEAKER: John Delaney, University of Washington
   TOPIC: “THE NEPTUNE PROJECT: AN INTERACTIVE EARTH-OCEAN
   OBSERVATORY AT THE SCALE OF A TECTONIC PLATE”
4:30 P.M. POSTER SESSION
6:00 P.M. EVENING FREE

Wednesday, March 27
8:00 A.M. CONTINENTAL BREAKFAST
9:30 A.M. TECHNICAL SESSION III: EARTH ENVIRONMENTS
   INVITED SPEAKER: Henk Meuzelaar, University of Utah
   TOPIC: “MAPPING AND MONITORING COMPLEX CHEMICAL COMPONENTS IN
   AMBIENT AIR USING FAST GC/MS AND MULTIVARIATE DATA ANALYSIS”
12:00 NOON INFORMAL LUNCH BUFFET / VENDOR EXPO
1:30 P.M. TECHNICAL SESSION IV: BIO-APPLICATIONS
3:00 P.M. TECHNICAL SESSION V: NOVEL CONCEPTS / MINIATURIZATION
   INVITED SPEAKER: Ara Chutjian, Jet Propulsion Laboratory
   TOPIC: “MINIATURE MASS SPECTROMETERS AND FRONT-END INTERFACES”
7:00 P.M. RECEPTION/CONFERENCE DINNER, COURTYARD OLD PASADENA
   GUEST SPEAKER: Dr. Charles Elachi, Director, Jet Propulsion Laboratory,
   “SPACE EXPLORATION IN THE NEXT DECADE”

Thursday, March 28
8:00 A.M. CONTINENTAL BREAKFAST
8:30 A.M. TECHNICAL SESSION I: MINIATURIZATION / TECHNICAL ISSUES
   INVITED SPEAKER: Phil Muntz, University of Southern California
   TOPIC: “THE TECHNICAL ISSUES ASSOCIATED WITH HIGHLY MINIATURIZED
   VACUUM SYSTEMS”
12:00 NOON INFORMAL LUNCH BUFFET
1:30 P.M. TECHNICAL SESSION II: COMMERCIALIZATION ISSUES
   INVITED SPEAKER: Peter Kardok, Alcatel
   TOPIC: “THE ISSUES LIMITING LARGE-SCALE COMMERCIALIZATION OF
   MINIATURE VACUUM SYSTEMS”
5:00 P.M. CLOSING REMARKS
5:30 P.M. ADJOURN

TRAVEL DAY

Monday, March 25
7:00 P.M. WELCOME RECEPTION AT COURTYARD OLD PASADENA