Liquid Metal Micro Heat Pipes for Space Radiator Applications

by

F. M. Gerner
Department of Mechanical, Industrial and Nuclear Engineering

and

H. T. Henderson
Department of Electrical & Computer Engineering

University of Cincinnati
Mail Location 72
Cincinnati, Ohio 45221-0072

NASA Grant # NAG3-1392
TABLE OF CONTENTS

Table of Contents .......................................................................................................... 2

I. PROJECT SUMMARY ............................................................................................ 3
II. INTRODUCTION................................................................................................. 4
III. FABRICATIONS OF LIQUID METAL MHP'S..................................................... 4
IV. BIBLIOGRAPHY.................................................................................................... 13
I. PROJECT SUMMARY

“Micromachining” is a chemical means of etching three-dimensional structures, typically in single-crystalline silicon. These techniques are leading toward what is coming to be referred to as MEMS (Micro Electro Mechanical Systems), where in addition to the ordinary two-dimensional (planar) microelectronics, it is possible to build three-dimensional micromotors, electrically-actuated microvalves, hydraulic systems and much more on the same microchip. These techniques become possible because of differential etching rates of various crystallographic planes and materials used for semiconductor microfabrication.

The University of Cincinnati group in collaboration with Karl Baker at NASA Lewis were the first to form micro heat pipes in silicon by the above techniques. Work is ongoing at a modest level, but several essential bonding and packaging techniques have been recently developed.

Currently, we have constructed and filled water/silicon micro heat pipes. Preliminary thermal tests of arrays of 125 micro heat pipes etched in a 1 inch x 1 inch x 250 μm silicon wafer have been completed. These pipes are instrumented with extremely small P-N junctions to measure their effective conductivity and their maximum operating power. A relatively simple one-dimensional model has been developed in order to predict micro heat pipes' operating characteristics. This information can be used to optimize micro heat pipe design with respect to length, hydraulic diameter, and number of pipes.

Work is progressing on the fabrication of liquid-metal micro heat pipes. In order to be compatible with the liquid metal (sodium or potassium), the inside of the micro heat pipes will be coated with a refractory metal (such as tungsten, molybdenum or titanium). Compatibility tests have been conducted in a high-temperature furnace. It has been shown that sodium reacts with silicon, silicon oxides and silicon nitride materials. Therefore, if sodium or potassium is used, the silicon will be coated with either tungsten, molybdenum or titanium. If mercury is used as the working fluid, bare silicon may be used. If a refractory coating is needed, a new bonding procedure must be developed to join these metals. Fusion and diffusion bonding processes are being developed for this purpose. It is expected that test results will demonstrate that the effective specific thermal conductivity of liquid-metal micro heat pipes is several orders-of-magnitude larger than for copper.
II. INTRODUCTION

Micro heat pipes are novel new devices capable of removing large amounts of energy over extremely small areas, i.e. high heat flux cooling devices, which is especially appropriate for cooling microelectronic chips and circuits. The basic design, which is shown in Fig. 1, is to form an array of micro heat pipes in silicon and use them as a very efficient thermal spreader. The procedure is to use photolithography and anisotropic wet-chemical etching on (100) oriented silicon to create triangular grooves of width 100 μm (the depth is comparable, since the etch angle is 54.7 degrees) and space them 100 μm apart. Bonding of a sheet of high temperature Pyrex glass, for water/silicon pipes, or a piece of silicon, for liquid metal/silicon pipes, is used to close the devices. They are then filled and sealed. The grooves may be coated with a refractory metal for enhanced protection against any possible degradation by the liquid metals.
III. Fabrication of Liquid Metal Micro Heat Pipes

In the past one year, a significant amount of work has been done on fabricating high temperature micro heat pipes in terms of temperature sensors, bonding and filling issues (low and high temperature). The fabrication issues and problems encountered are discussed in the following section.

Fabrication of Polysilicon Temperature Sensors

In order to test the micro heat pipes, temperature profiles are necessary. The P-N junctions fabricated in the past can be operated only at low temperatures in the temperature range of 20°C to 200°C (which is in the operating temperature range of water heat pipes). But as the operating temperature goes up (as required for mercury micro heat pipes) the diodes cannot be used as temperature sensors and other types of temperature sensing elements are required. To test the high temperature micro heat pipe, polysilicon temperature sensors have been successfully fabricated showing a very high sensitivity of .005 kΩ/°C. Several mask levels were required to fabricate these sensors. Test runs were required to get the necessary parameters such as the deposition time, annealing temperature and doping concentration. More tests are being performed to increase the sensitivity by an order of magnitude.

Fabrication steps for polysilicon temperature sensors are as follows: The wafers are first oxidized after which polysilicon is deposited by LPCVD (low pressure chemical vapor deposition) at a temperature of 590°C with a pressure of milli torr. Various thicknesses of .25μm, .54μm, and 1μm polysilicon have been deposited for sensitivity measurement. The deposited polysilicon is then annealed at different temperatures to allow the grain boundaries in polysilicon to grow. The polysilicon is then doped with boron and patterned by lithography process. The patterned polysilicon resistors are then RIE (reactive ion etching) etched by using SF₆ plasma. The photoresist acts as an etch barrier on polysilicon (to define the resistors). The etch rate of the polysilicon had to be characterized first and the etch rate was found to be about .1μm/min. After the polysilicon etch the wafers are oxidized to grow a very thin layer of SiO₂ to passivate the polysilicon surface. The wafers are then patterned for metallization of 100A of titanium and 8000A of aluminum. The fabricated polysilicon sensors are profiled for temperature sensing in an environmental chamber. Fabrication steps necessary for high temperature micro heat pipes incorporating the polysilicon temperature sensors are shown in Fig. 2. Figure 3 shows different types of polysilicon resistors with different resistances fabricated in order to determine which gave the maximum sensitivity. Figure 4 shows a curve of resistance vs. temperature for one type of device.

In our earlier work, 100μm wide pipes with a spacing of 100μm had been fabricated. A new mask has been designed for fabricating 250μm wide micro heat pipes with a spacing of 100μm between the pipes, to explore the difference in operation between the 100μm and 250μm. The 250 μm micro heat pipe has been successfully fabricated. Figure 5 shows a picture of the
100 μm and 250 μm wide heat pipes. Six different mask levels have been used for fabricating and instrumenting the 250μm micro heat pipes.

Figure 1 - Layout of Micro Heat Pipes on a Silicon Wafer
These masks include fabrication of the diodes and polysilicon temperature sensors on the 250μm wide pipes. The masks can also be used for instrumenting the existing low-temperature micro heat pipes. Diode and polysilicon temperature sensors can be fabricated using the same six mask levels. During the past six months, problems associated with boron diffusion (for p-type) have been solved. Polysilicon temperature sensors have been successfully fabricated and tested. Improvements on sensitivity issues are being pursued.

**Bonding Experiments**

The thermal conductivity of the 7740 Pyrex glass which is electrostatically bonded to cap the micro heat pipes is 0.1 w/m°C and that of silicon is 80 w/m°C. Calculations performed, based on the thermal resistance network, showed that a 30 mil thick glass wafer used for electrostatic bonding has a thermal resistance of only four times that of silicon. So to increase the thermal resistance of glass (i.e. to minimize the heat losses in glass) a 15 mils thick 7740 glass wafer has been specially fabricated and electrostatically bonded to silicon. Another option to minimize the heat losses in glass was to use quartz instead of 7740 Pyrex, since the thermal conductivity of the 7740 glass is approximately 10 times that of quartz. Experiments have been conducted to test electrostatic bonding of quartz to silicon. The two bonded very well at high temperatures above (500°C). But as the temperature was brought down, due to change of phase of quartz and the difference in thermal coefficient of expansion, the bonded wafers cracked. Bonding of quartz and silicon may also be accomplished by fusion bonding.

Experiments are being conducted to see if bonding can be accomplished by using a silicon dioxide layer between the quartz and silicon. Another way to bond quartz to silicon is by using polyimide, which has been conducted. There are problems associated with the latter method in terms of the uniformity of the bond across the wafer. Experiments to get better uniformity, are also in progress. Silicon-to-silicon fusion bonding has also been successfully accomplished. An IR system has been set up to view the bonding interface.

**Compatibility Test**

For the micro heat pipes it is necessary that the working fluid inside the pipes does not react with the base material or the capping material. In the past it has been shown that sodium reacts with silicon, silicon dioxide and silicon carbide. Refractory metals such as chromium, tungsten and molybdenum have been e-beam evaporated on the surface of a silicon wafer to protect the surface of silicon. These layers are very thin in the order of 1000 A and therefore tend to have pinholes. When tested as a protective coating for sodium it was seen that sodium penetrated through the pinholes in the refractory metal and reacted with the underlying silicon.

To get a good surface finish these metals can be electroplated. Mercury is another working fluid which can be used instead of sodium. Compatibility tests using mercury as a working fluid are being done. It has also been shown that glass does not react with Hg at a
temperature of 450°C. A vacuum system has been set up to show that Hg does not react with silicon.

Figure 2 - Fabrication Steps of Liquid Metal MHP
Resistance vs. Temperature
wafer no. 28 Device type 6 (6d,3r) file poly9

Figure 4 - Typical Resistance Versus Temperature Curve of Polysilicon Sensor
**Fitting/Testing**

In the past a copper plate has been used for making connection from the pipes to the vacuum system to evacuate and fill the pipes. This copper plate was glued on to the silicon wafer which caused leakage of vacuum inside the pipes; and also at an elevated temperature of 120°C the attached copper plate came off the silicon surface because the glue used to seal the copper plate did not hold at elevated temperatures. In order to form a better seal, experiments were conducted for using a glass tube "nipple" (7740 Pyrex) instead of the copper plate. A one inch long and 3 mm diameter (outer) 7740 glass tube was taken and one end of the pipe was flared out and then the base of the flared end was electrostatically bonded to silicon. The tube end was attached to the vacuum system by tygon tubing. Some improvement is needed, as these glass nipples are locally made so the surface finish of the flared end is not smooth or optically flat, which is necessary for good electrostatic bonding. These nipples were therefore polished using a 5μm alumina grit and were examined for optical flatness using a sodium lamp as an interferometer. Also an optical microscope was used to check the roughness of the flared surface. A jig has been prepared for polishing these nipples. Experiments need to be conducted to find out what flatness and optical clarity is necessary for bonding the nipple. Figure 6 shows glass nipples being bonded to a silicon substrate.

A printed circuit board is also being made on quartz with gold lines, because the ordinary PCB's can be used only in the military range not in the range of 400°C. Preliminary work, to explore the feasibility of the quartz PCB, has been successfully done. The silicon wafer will then be mounted on this PCB using polyimide.

The aluminum bonding pads will be wire bonded to the gold lines on the quartz PCB. First the quartz plate will be patterned and then molten 14 k gold will be put on the glass plate using a diamond tip and the plate will be baked at a temperature of 600°C to form solid gold lines. Silver electrically-conducting epoxy will then be used to attach the regular electrical wire to the plate, which in turn will be connected to the data acquisition system.
Figure 5 - Photographs of Heat Pipes of Various Widths
Figure 6 - Photographs Showing Glass "Nipple" Used for Filling
IV. Bibliography


