RAPID LOW TEMPERATURE BONDING OF SILICON TO STEEL FOR MEMS SENSORS

Andrew Cao
Department of Mechanical Engineering
Berkeley Sensors and Actuators Center
University of California at Berkeley

Brian Sosnowchik
Department of Mechanical Engineering
Berkeley Sensors and Actuators Center
University of California at Berkeley

Liwei Lin
Department of Mechanical Engineering
Berkeley Sensors and Actuators Center
University of California at Berkeley

Albert Pisano
Department of Mechanical Engineering
Berkeley Sensors and Actuators Center
University of California at Berkeley

ABSTRACT
We have developed a rapid silicon to steel bonding process used to attach silicon microsensors onto steel. The specific application is to bond silicon based strain sensors to steel structures for real time strain sensing. Steel samples are electroplated with eutectic Pb/Sn alloy (Mp 183°C) and are bonded to silicon dice coated with Cr/Au or Cr/Cu/Ni/PbSn layers. Bonding took place in the inert environment of a rapid thermal annealing (RTA) oven at 220°C for about 20 seconds, which caused minimal damage to the steel samples. The silicon to steel bond survived >1000µε of static loading at the steel surface and 1000 cycles of >1000µε cyclic strain at the steel surface without failure. Corrosion resistance of the silicon to steel bond was tested by immersion in various oils and salt solutions, and the bonding method thus far has been successful.

INTRODUCTION
The ability to bond silicon to steel has many potential engineering applications as it allows the integration of micro-sensors onto various structures. Steel is one of the most commonly utilized structural metals, used extensively form buildings and vehicles to machines and components. Additionally, many micro-sensors, including accelerometers, temperature sensors, pressure sensors, and strain gauges, are built using silicon micromachining processes. Even if the device itself is not made from silicon, the sensors are often microfabricated on top of a silicon substrate. Hence the ability to bond silicon sensors to steel would allow for the monitoring of many structures in real time. The goal of this research is to develop a rapid silicon to steel bonding process that will not harm previously tempered steel, and that can survive in harsh environments for several years. A micro resonant strain gauge has been previously developed our research group [1]. This silicon-based strain gauge can be bonded on to steel structure to monitor strain in real time as shown in Fig. 1.

A silicon to steel bonding process suitable for industrial use must satisfy several important criteria. The bonding process must be fast; it should take no more than several tens of seconds. The bonding temperature must be low, such that there would be minimal heat affected damage and thermally induced strain. The silicon to steel bond must be strong enough to withstand millions of cycles of high stress and strain without failure. The bond must survive for years in the corrosive environment of the outdoors and in oily machinery. And it must survive many cycles of temperature fluctuation without delaminating.

Figure 1. A micro resonant strain gauge bonded on to a piece of steel for real time strain sensing applications.
Many common methods of steel bonding would not be suitable for various reasons. Steel begins to lose its tempering around 230°C; hence a suitable bonding method must remain below this temperature. Welding and brazing (>425°C) form excellent mechanical bonds, but the high temperature involved are likely to cause unacceptable heat damage and residual stresses. Solid-state diffusion bonding and eutectic bonding [2,3,4] can be achieved below the melting temperatures of the materials involved, but these processes take from several minutes to hours. Adhesive bonding is a very low temperature process, but the cure time is in the tens of minutes. Polymers also don’t have suitable mechanical characteristics and corrosion resistance. Ultrasonic and thermosonic bonding are fast, low temperature bonding processes [5,6,7]. However the mechanical shock and vibration in these bonding processes might damage suspended MEMS structures, and hence they have not been investigated in this research.

This research investigates a fluxless soldering process [8,9,10,11,12] to bond silicon to steel. The solder used is the eutectic tin lead alloy (37/63 Pb/Sn), which has a melting temperature of 183°C. The time required for this solder to wet a suitable metal and form a good bond is typically several seconds. Steel samples are electroplated with Pb/Sn solder, and bonded to either gold or nickel layers deposited on to silicon.

![Figure 2. Schematic of steel sample preparation. 1) Steel samples are cleaned on the front ("F" side) of the parallel and 2) masked with polyimide tape on the front and back ("B" side). 3) They are placed into the plating solution, to the point where the plating solution rises to the level of the tape on the front side. 4) When the plating is finished, the tape is removed from the steel.](image-url)

**FABRICATION OVERVIEW & SAMPLE PREPARATION**

The bonding for this work involved two components: preparation of the steel samples and fabrication of the silicon test dice.

**Steel Sample Preparation**

Spring steel parallels of length 6in, width 1.25in, and thickness 0.03125in (1/32in) were used for the study, and the following conditioning process is illustrated in Fig. 2. The steel parallels were precision ground to remove the steel’s native oxide layer and to condition the surface to a roughness of around 2µm peak-to-peak. The steel was then cleaned with acetone, masked with 0.0025in polyimide film tape (shown as white in Fig. 2), and placed into a bath of 37/63 Pb/Sn electroplating solution (Techni Solder Matte NF 820 HS 60/40, Technic Inc., Anaheim, CA). The polyimide tape served two purposes during the plating process; first, it served as a successful masking layer for the backside of the parallel, as well as providing an effective means by which to characterize the thickness of the plated layer. The plating was performed at a rate of 18.8 milliamps per square inch for approximately 3.8 hours, which resulted in an average thickness of approximately 40µm.

After the plating process, the Pb/Sn layer atop the parallels was then reflowed via infrared heating in a Heatpulse 210T Rapid Thermal Annealing (RTA) apparatus [13,14,15]. The annealing furnace contains thirteen 1.5 KW halogen lamps, which are arranged in upper and lower banks of 6 and 7 bulbs respectively. The lamps are housed in water-cooled, reflective walls, and a hermetically sealed quartz annealing tube is positioned between the banks. The visible light from the continuous-wave (CW) lamps passes through the quartz annealing tube and wafer tray and is absorbed by the sample. The quartz tube can be purged with inert gas so the annealed material will not oxidize during high temperature process, and the temperature inside the chamber is measured using a thermocouple.

![Figure 3. Temperature profile inside the RTP bonding chamber; ideal heating profile consists of a 20 second ramp up to 220°C, a 20 second hold at 220°C, and an instant cool down to room temperature. The actual temperature in the chamber takes about 80 seconds to fall below 80 °C.](image-url)
in the temperature to 220°C, where it remained steady for another 20s. In practice, the chamber temperature deviated slightly due to the control mechanisms of the apparatus. Moreover, due to the nature of the equipment, the cooling of the chamber was not immediate, as the ideal profile shows it, but rather, the cooling was performed by forced convection of heat from the steel to the nitrogen environment and took approximately 80 seconds for the chamber to reach a temperature of 80°C.

Silicon Test Dice Preparation

In order to bond silicon to steel, thin metal layers were deposited on the silicon, since Pb/Sn solder does not wet to Si, or more appropriately, to the native SiO$_2$ inherently present on a silicon surface. Two different silicon test chips were used in the experiments—one with a gold underside, and one with a nickel underside. They were fabricated as follows:

1. **Gold (1000Å) on a 550µm chip**—A four inch, 550µm, p-type wafer of <100> orientation was first Piranha cleaned at 70°C for 10 minutes to remove organic residues. Then, using a Veeco 401 Vacuum System, a 200Å layer of chromium (R.D. Mathis Co., 99.9% min pure) was first evaporated onto the wafer at a rate of approximately 0.5Å/s, followed by a subsequent 1000Å deposition of gold (Refining Systems Inc., 99.99% pure) deposited at a rate of approximately 3Å/s. Following the evaporation of the Cr/Au layer, the sample was annealed in argon at 160°C for 30 minutes.

2. **Nickel (1.5µm) on a 550µm chip**—A four inch, 550µm, p-type wafer of <100> orientation was first Piranha cleaned at 70°C for 10 minutes to remove organic residues. Then, using a Veeco 401 Vacuum System, a 500Å layer of chromium (R.D. Mathis Co., 99.9% min purity) was first evaporated onto the wafer at a rate of approximately 1Å/s, followed by a subsequent 5000Å deposition of copper (Sigma-Aldrich, 99.99% pure) deposited at a rate of approximately 5Å/s. Following the evaporation of a Cr/Cu conductive layer, the wafer was immediately submerged in a bath of Techni Nickel S® nickel electroplating solution (Technic Inc), and a 1.5µm layer of nickel was plated onto the wafer. Following the nickel plating step, a 1.25µm cap of 37/63 Pb/Sn was immediately plated to the nickel in order to prevent the nickel from oxidizing.

Silicon-to-Steel Bonding in Rapid Thermal Annealer

In order to prepare the steel samples for testing, the steel samples underwent the following fabrication sequence. First, the Pb/Sn layer was deposited onto the steel per the aforementioned method. After performing heating cycle from Fig. 3 required to reflow the solder and a small time for the parallel to return to an adequate handling temperature, the parallels were then removed from the RTA equipment and allowed to cool to room temperature while silicon test dice were placed onto the top of the solder with the thin film layer facing the Pb/Sn layer. The parallels were then carefully loaded back into the RTA machine and the thermal cycle illustrated before was performed. Again, after a short delay necessary to allow the parallels to return to an appropriate handling temperature, the parallels were removed from the RTA chamber with the silicon test dice bonded to the steel.

**EXPERIMENTAL SETUP**

Three different tests have been performed on the bonded samples; static bond strength tests, fatigue strength tests, and...
corrosion resistance tests. For the static and fatigue experiments, the test was performed using a 4-point bending jig fixture. The parallels tested had a thickness of 1/32in, and a 40µm thick layer of Pb/Sn alloy plated where the silicon dice were bonded. After the silicon dice were bonded to steel parallels, foil strain gages were then applied to the top of the silicon chips as shown in Fig. 4. The foil strain gages used had a gage length of 1.57mm and a width of 3.05mm (Vishay Micro-Measurements, Raleigh, NC) and were used to measure the strain at the surface of the 5mm² silicon chips. Additionally, Vishay M-Bond 610 was selected as the adhesive for the foil strain gages because of its specified ability to be deposited at thickness of 0.0002in and less, while remaining hard and void-free.

The purpose of applying the strain gages to the top of the silicon chip was to determine when a bonding failure “event” had occurred. Prior to performing the static and fatigue tests, numerous qualitative tests were undertaken. It was observed that failure at high surface strains occurred by fracture of the silicon chip or delamination of the bond. However, while the degree of delamination could range from complete to minimal, it could not be accurately determined at what strain the delamination and/or fracture of the silicon. Hence, by placing a strain gage to the surface of the silicon, even though the precise value of the strain and stress at the Pb/Sn-Silicon interface could not be measured, the output from the foil strain gage vs. time and load vs. time plots can be used to determine the theoretical strain at the surface of the steel. Test samples were loaded in a 4-point bending fixture as shown in Fig. 5. Two silicon dice were bonded at the center of the parallel between the two inner loading points in the 4-point bend test. This area has a uniform bending moment but no shear stress. An increasing load was applied using an Instron® tensile testing machine until an “event” occurred. The strain at the surface of the bond was calculated as follows:

\[
\sigma_{\text{max}} = \frac{PLt}{24I_{xx}} \quad (1)
\]
\[
\varepsilon_{\text{max}} = \frac{PL}{12EI_{xx}} \quad (2)
\]

where \(E\) is the Young’s modulus of the material, \(I_{xx}\) is the area moment of inertia, \(\sigma_{\text{max}}\) is the maximum stress, and \(\varepsilon_{\text{max}}\) is maximum strain. The location of the maximum stress and strain is also where the silicon die is bonded on the steel.

Static Tests

Static tests were performed by ramping the load from 0lbs to 175lbs over a period of approximately 180 seconds, with a delay at the beginning of the test and again when the loading had reached 175lbs. Failure was determined by the point at which an “event” occurred.

Fatigue Tests

Fatigue tests were performed using the same 4-point bending setup with a load of ~26-28lbs (depending on the steel sample width), corresponding to about 1800µε at the steel/silicon interface. One thousand cycles of strain were applied on each parallel sample with two silicon dice bonded on the surface. The strain at the silicon surface was measured using a foil strain gauge affixed to the surface of the silicon test dice.

Corrosion Resistance Test

The corrosion resistance test was performed to determine the deleterious effects of exposure of the bond to extreme environmental temperatures and conditions. For the test, various different fluids were selected that might be found present in an outdoors environment. They were the following:

- DI water at 85°C
- DI water saturated with iodized salt at 85°C
- Valvoline® High Performance Gear Oil 85W-140 at 85°C
- Peak® Antifreeze at 85°C
- Havoline® 5W-30 Motor Oil at 100°C
- Havoline® 20W-50 Motor Oil at 100°C
- Pennzoil® DexronIII-Mercon Transmission Fluid at 100°C
- Pyroil® Power Steering Fluid at 100°C

The fluids were placed into Pyrex beakers, wrapped with insulation to reduce thermal gradients and placed onto a hotplate. Two hotplates were used and the temperatures selected for the hotplates were determined and set several tens
of degrees below the lowest flash point temperature of the fluids on each hotplate. When the experiment began, there were concerns about thermal gradients across the top of the hotplate, but after initiating the long-term corrosion resistance test, it was found that the temperature was very uniform across the hotplate.

The steel parallels for the corrosion resistance test were all prepared under the fabrication sequence detailed above, and three of the gold and nickel chips were bonded to the parallels. After the chips were then submerged in the fluids, they were checked daily to observe failure of the dice.

RESULTS

Silicon dice bonded to steel using either the Au-to-solder or Ni-to-solder system show promising results. In the static loading test, the dice can survive over 1000µε at the surface of the silicon before failure. Figures 6 & 7 show the plots of static loading vs. measured strain at the silicon die surface for gold and nickel, respectively. For the gold sample, as loading was increased, the measured strain at the silicon surface increased linearly until the die failed by cracking. The failure point was indicated by jump in measured strain. The surface defect of the silicon die is likely to have a large effect on the point of failure; hence there is a big difference from die to die where failure occurs. However, the slope of the strain vs. applied load is fairly constant, which suggest the silicon and steel has formed a good bond.

Silicon to steel bonds survived short-term fatigue test of 1000 cycles without failure. The silicon dice were cycled at 50µε to 700µε at a frequency of 1Hz. A plot of strain at Si surface vs. stress cycle is shown in Figs. 8 & 9. Notice in Fig. 8 that there is a gradual decrease in the maximum measured strain during the course of the test. This decrease could be caused by creep of the solder at the silicon to steel bonding interface or creep of epoxy glue at the silicon to strain gauge interface. Additionally, notice that in Fig. 9, there is a gradual reduction in both the peak and minimum strain as the test progressed. When this sample was unloaded, the result was a residual strain at the surface of the silicon of approximately 84µε in compression, which could also be the result of the solder creeping. This effect will be investigated in the future; however, all samples with gold and nickel adhesion layers survived the short term fatigue test with no signs of failure.

Finally, at the time of the submission of this paper, the corrosion resistance test has been established for over six weeks, and all of the Au-to-solder and the Ni-to-solder samples have survived the aforementioned corrosive fluids, with the exception of one nickel sample in the transmission fluid, which was damaged during the test setup and failed not as a result of the fluid. Qualitative observation and testing will continue for the corrosion resistance test, and future investigations will focus on combining corrosion testing with low-strain static or fatigue testing to determine the detrimental effects of the fluids.

---

**Figure 6.** Silicon dice with gold adhesion layer bonded to steel can survive over 1000µε before failure occurs.

**Figure 7.** Silicon dice with nickel adhesion layers bonded to steel. While one chip fractured (sample 2) and the other delaminated after a while (sample 1), both survived 1000µε before the “events” occurred.
CONCLUSION

We have demonstrated a rapid fluxless soldering process to bond silicon dice on to steel. This bonding process takes less than 60 seconds and causes minimal damage to steel. The silicon to steel bonds have both good mechanical characteristics and corrosion resistance. As such, based upon the tests performed so far, it promises to be a good bonding method to attach silicon-based microsensors onto steel for real time sensing of strain or other parameters.

FUTURE WORK

Future research will focus on the use of lead free solder with improved mechanical characteristics and corrosion resistance. As such, based upon the tests performed so far, it promises to be a good bonding method to attach silicon-based microsensors onto steel for real time sensing of strain or other parameters.

ACKNOWLEDGMENTS

The project is supported by the army research office (ARO) grant DAAD 19-02-1-0198. Process development for this research is performed at the U.C. Berkeley Microlab. The authors would like to acknowledge the help of Dr. Anand Jog, Mr. Robert Azevedo, and the UC Berkeley Mechanical Engineering Machine Shop.

REFERENCES


Figure 8. Fatigue test of silicon to steel bond using gold adhesion layer. Notice there is a small gradual decrease in maximum strain output, but the test resulted with almost no residual stress transmitted when returned to the non-loaded position.

Figure 9. Fatigue test of silicon to steel bond using nickel adhesion layer. Notice there is a small gradual decrease in peak and minimum load Si surface strain, which results in a difference between the starting and finishing Min-Load Strain Output, and a residual output for the zero-load condition.