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METALLIZATION AND PHOTOLITHOGRAPHIC
PROCESSES AND PROCEDURES FOR MC2730
RTG THERMOPILE INTRACONNECTIONS

MASTER

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METALLIZATION AND PHOTOLITHOGRAPHIC PROCESSES AND
PROCEDURES FOR MC2730 RTG THERMOPILE INTRACONNECTIONS

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March 1974

ABSTRACT

Processes and procedures have been developed for applying the thin film tungsten electrical intraconnections to the MC2730 RTG "one-dimensional" thermopile after polishing, the surface to be metallized is cleaned with a detergent/organic solvent procedure and then etched with hydrofluoric acid to minimize the oxide. Tungsten contacts are sputtered onto the thermopile and the individual contacts are photolithographically defined using a negative acting photoresist in conjunction with a potassium ferricyanide etchant. The processes detailed in this report were used to process 89 thermopiles with an 80% effective yield.

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INTRODUCTION

The 1D thermopile contains two channels, both electrically independent of each other which are separated by a .004" thick glass insulating layer. Each channel contains 22 p-type and 22 n-type elements, consisting of 80/20 at.% SiGe polycrystalline alloy composition, approximately .007" thick. These elements are separated from each other by .001" thick glass insulating layers. The thermopile is assembled by a hot press laminating process. Overall dimensions of the thermopile are .351 x .284 x 1".

The elements of each channel are connected in series by tungsten contacts. Tungsten is sputtered to a thickness of 3μ and the contact areas are defined by a photolithographic process. The output connections are made by attaching gold ribbon leads to the thermopile by a eutectic die bonding and parallel gap welding process.^{1,2} The element interconnection pattern for the top (hot end) and bottom (cold end) of the thermopile is shown in Figure 1. The hot end contact pattern consists of 22 contacts whereas the cold end pattern has only 21 contacts since the output leads are connected to the outer elements. The 4-point probe method used for measuring interconnection resistance and the effects of process variables on contact resistance are covered in reports by J. N. Sweet.^{3,4}

This report describes the metallization and photolithographic processes used to provide tungsten interelement contacts to 1D thermopiles. The purpose of this report is to document these processes and procedures as they stood on March 1, 1973, when contact process responsibility was transferred to Division 2334. Detailed procedures are listed in the Appendices.

FIGURE 1. Interelement Connection Pattern for the Hot End and the Cold End of a Thermopile.

SURFACE PREPARATION AND CLEANING

As received from the glassing operation, the thermopiles are lapped and polished followed by a chemical cleaning process to provide a suitable surface for tungsten metallization and photolithographic definition of good adherent and low electrical resistant contacts. These processes are described in a report by Traeger and Knauss.⁵

Briefly, the thermopiles are hand lapped using 1800 grit aluminum oxide to establish the final overall dimensions, followed by hand polishing the ends using subsequent application of 6 μ , 1 μ , and $\frac{1}{2}\mu$ diamond paste. After polishing, the thermopiles are rinsed in alcohol, wrapped in lens tissues and placed in plastic containers for safe handling.

Gross contaminants are removed from the thermopiles by scrubbing all surfaces in trichloroethylene using a cotton bud, followed by scrubbing in methyl alcohol. Immediately prior to metallization, the thermopiles undergo a thorough cleaning in subsequent ultrasonic baths of trichloroethylene, a detergent solution, and deionized water. Oxide formation on the surfaces to be metallized is reduced by an HF etch, ultrasonically rinsed in deionized water, and isopropyl alcohol and immediately loaded into the sputtering system and vacuum pumped. Detailed cleaning procedures are described in Appendix A.

THERMOPILE METALLIZATION

Metallization of the 1D thermopile consists of dc sputtering tungsten on both ends of the thermopile for the interelement connections. This is a two step sputtering process.

The sputtering system consists of a Welch 260 liter/second turbomolecular pump with a Welch Model 1397 forepump and a bakeable stainless steel bell jar with a Viton seal to the vacuum system base plate. The argon gas flow is controlled with a Granville Phillips leak valve and the vacuum chamber pressure is controlled with a throttle valve in the throat of the base plate. The sputtering gas used is commercial grade argon passed through a Mathis GP-100 gas purifier. The sputtering module is a custom designed two target system fitted with a special thermopile fixture mounted on a rotatable anode plate.⁶ A sketch of the sputtering module is shown in Figure 2. The photograph in Figure 3 shows the thermopile fixture in position under the target.

The thermopile metallization fixture, shown in Figure 4, is capable of holding 24 thermopiles. The center position is occupied by a chromel-alumel thermocouple element mounted in a stainless steel block representing a thermopile. The thermocouple element mounted in a stainless steel block representing a thermopile. The thermocouple element is inserted into a hole in the block from the underneath side of the thermopile fixture and held captive close to the upper end of the block with a set screw. The fixture is heated by means of a Thermocoax[®] heating element brazed into grooves located in the side of the individual sections of the fixture. The design of the fixture is such that it can be disassembled for periodic cleaning.

Within minutes after cleaning, the thermopiles are placed into the sputtering fixture with teflon coated tweezers. Polyethylene gloves are worn by the operator to protect the sputtering furniture from contamination during loading operations.

Each thermopile is identified by a number inscribed on its side. As the number is read, the left hand end of the thermopile has been designated as the "cold end." The right hand end is the "hot end." For the first end metallization, the thermopiles are placed into the sputtering fixture with the cold end facing up.

[®]Trademark, Amperex Electronic Corporation.

FIGURE 2. A Sketch of the Sputtering Module

FIGURE 3. Thermopile Fixture in Position Under Target

FIGURE 4. Thermopile Metallization Fixture

The thermopiles rest on pads placed on top of stainless steel blocks. The purpose of these pads is to prevent abrasion damage to the polished surface by the vacuum system vibrations, and to reduce contamination of the hot end surface during the first end metallization. These pads are made of Corning 7059 glass and are subjected to the same cleaning procedure as the thermopiles except for the HF etch step.

The exposed thermopile ends are aligned with the top surface of the fixture by means of machine screws located at the bottom of the fixture and secured in position with locking nuts as shown in Figure 5.

A glass control sample is included in each deposition run for the purpose of film thickness and film resistivity evaluation.

All vacant positions in the fixture are filled with stainless steel blocks. The top surfaces of these blocks have been previously coated with tungsten to prevent stainless steel contamination of the thermopiles during initial stages of sputtering. Following loading, the thermopile fixture is positioned under the shutter by rotating the anode plate. The bell jar is lowered and the pumping procedure started.

The vacuum chamber is rough pumped to a pressure of 10 Torr at which time the turbomolecular pump is started. After the pressure in the chamber reaches 2×10^{-5} Torr, or less, the bell jar, the thermopile fixture, and the turbomolecular pump heaters are turned on. The bell jar heating element power is approximately 1100 watts (115 Vdc at 9.6 A). The bell jar bake period is 8 hours (timer controlled) and the average temperature is 150°C controlled by a thermocouple sensor. The thermopile fixture heating element power is 106 watts (24 Vdc at 4.4 A) and the temperature is monitored with a Doric Model 300 temperature indicator. The turbomolecular pump bake period is 60 minutes controlled by a Cramer timer.

FIGURE 5. Thermopile Lowered Into Fixture

Loading and starting of the vacuum system is normally scheduled for the latter part of the work day so that the bell jar bake and cooling periods take place during the night hours.

The following morning, the ultimate pressure should be at $2 - 4 \times 10^{-7}$ Torr. The sputtering procedure is started by turning on the gas purifier heater and the Hastings flow rate meter, allowing 15 minutes for both instruments to stabilize. Argon is then admitted into the chamber and the leak valve and throttle are adjusted to provide a flow rate of 145 SCCM at a pressure of 20 millitorr as indicated on the pressure gauge.* Following this, the high voltage to the sputtering target is applied, limiting the current to 50 mA. The sputtering voltage at this time should be between 3200 and 3500 V. During the 30 minute target clean-up period, the voltage is gradually increased to 4500 V, not exceeding 50 mA. The current will decrease and stabilize at 38 to 40 mA as the sputtering target temperature reaches equilibrium. Following the pre-sputtering period, the thermopile fixture is positioned under the target for 3 hours, during which time approximately 3 microns of tungsten are deposited. At the end of the 3 hour sputtering period, the high voltage to the sputtering target and the thermopile fixture heater power are turned off, and the thermopile fixture is rotated back under the shutter plate to protect the thermopiles from tungsten flake deposits. (During sputtering, tungsten is deposited on the surrounding furniture as well as on the thermopiles. Consequently, the built-up tungsten film flakes off under stresses of thermal cycling after several sputtering runs.) The argon is turned off, the throttle valve is opened to maximum throughput position, and the gas purifier heater power is turned off. The system is left pumping for the remainder of the day, allowing the thermopile fixture to cool as long as possible before completely shutting down the system.

*The Pirini gauge is calibrated for air. Argon pressure at this setting is 31 millitorr.

The system is shut down at the end of the day by turning off the power to the forepump and the turbomolecular pump. The foreline valve closes automatically when the pumps are turned off. The vacuum chamber is vented with argon to approximately 500 Torr. A slight pressure differential will keep the vacuum chamber sealed from room ambients until the following day.

On the third day, the thermopiles are turned over in the fixture by inserting a wire probe into a hole provided in the height adjustment screw, thereby lifting the thermopile high enough to grasp with tweezers. The glass samples used for resistivity and thickness measurements are replaced with clean ones. The thermopile fixture is rotated under the shutter, the bell jar lowered, evacuated and the bake and sputtering procedure repeated as before. This completes the tungsten metallization of the thermopiles.

The sputtering parameters used in this procedure are as follows:

Cathode (tungsten target) Diameter	6 in. (15.2 cm)
Cathode-Substrate Spacing	2.5 in. (6.3 cm)
Thermopile Fixture Temperature	
During Bakeout	365°C
During Sputtering	350° to 453°C
Presputtering Time (target cleanup)	30 minutes
Ultimate Pressure	2×10^{-7} Torr
Bakeout Time (bell jar)	8 hours
Bakeout Time (turbomolecular pump)	60 minutes
Bell Jar Bakeout Temperature	150°C
Argon Pressure	30 millitorr
Argon Flow Rate	145 SCCM

A step by step sputtering procedure is included in Appendix B.

The sheet resistance of the tungsten films deposited on the glass control sample is measured with a four point probe. A portion of the film is etched away to provide a step to measure the thickness using the Dektak[®] instrument. From the sheet resistance and the film thickness measurements, the resistivity is computed from the relationship,

$$\rho = dR_s ,$$

where, d is the thickness in cm, and R_s is the sheet resistance in ohms/square. The resistivity is generally less than $40 \mu\Omega\text{-cm}$. The thermopile temperature of a typical sputtering run shown in Figure 6 was measured with a thermocouple mounted in a stainless steel block representing a thermopile. During the bakeout period, the temperature reached 365°C , dropping to 350°C in the 30 minute target cleanup period. The temperature rise to 465°C during the sputtering period is related to 3 hours of sputtering at 4500 V, 40 mA, and 30μ argon pressure.

PHOTOLITHOGRAPHY

Contact areas on both ends of the thermopile are defined by a photolithographic technique. A 22 contact pattern is used for the hot end, and a 21 contact pattern for the cold end.

Photographic masks are prepared from patterns generated with the aid of a computer program.⁷ The 20:1 masters made on a Gerber plotter are then photo-reduced onto 2" x 2" glass plates with a Dekacon camera. The Gerber masters are prepared by Organization 7624 and the photo-reduction is done in Organization 7333. Due to non-uniformity in the glassing dimensions of the thermopile, a combination of several

[®]Trademark, Sloan Instruments Corporation.

FIGURE 6. Thermopile Fixture Temperature Profile

mask patterns must be used to achieve proper mask alignment during exposure. A set of mask patterns used for both ends of the thermopile is shown in Figure 7. The mask employing the combination of four strips with a 2-mil border dimension proved to be the most useful pattern. The interior of the rectangles representing the contacts are clear, and the remainder of the glass plate area is opaque. The large rectangles on either side of the contact pattern are used for locating the sides of the thermopile during alignment.

A negative acting KMER[®] photoresist used in printing the contact pattern is prepared by diluting 2 parts of KMER in one part of KMER thinner and is filtered through a Millipore[®] 14 micron filter. The filtering procedure is included in Appendix C. Because of limited storage life, a fresh supply of photoresist is prepared weekly in a brown eye dropper bottle and stored in a light tight container.

The initial steps in the procedure are to photodefine the contacts by coating the ends of the thermopile with photoresist and exposing the surfaces to a light source through an appropriate mask. An outline of this procedure is as follows:

1. Coat the surface with KMER photoresist.
2. Spin at 3000 rpm for 30 seconds.
3. Prebake in air at 110^oC for 10 minutes.
4. Expose through appropriate mask for 30 seconds.
5. Repeat Step 4 with proper mask as required.
6. Develop using spray KMER developer for approximately 25-30 seconds.
Leave wet.
7. Spray with isopropyl alcohol and blow dry.
8. Postbake at 150^oC for 30 minutes.
9. Store in a clean, dust free area.

[®] Trademark, Eastman Kodak Co., Rochester, New York, "Kodak Metal Etch Resist."

[®] Trademark, Millipore Corp., Bedford, Mass., Catalog No. NCWG04700.

FIGURE 7. A Set of Exposure Mask Patterns

Photoresist is applied to the thermopile surface by a spinning technique. The spinning apparatus shown in Figure 8 is fitted with a special fixture, (see Figure 9) to hold the thermopile during the spinning operation. The alignment and exposure operation is done on a Westinghouse alignment machine fitted with a special thermopile holder. The alignment and exposure equipment is shown in Figure 10. Mask alignment is done by bringing the mask in near contact with the photoresist coating on the end of the thermopile with the aid of the microscope. After aligning the mask to the proper elements on the thermopile, the mask is then brought into contact with the photoresist and the exposure is made with an ultraviolet light which polymerizes the exposed photoresist.

After exposure, the photoresist is developed by alternately spraying the ends of the thermopile with KMER developer from 25 to 30 seconds, followed by a spray rinse with isopropyl alcohol and blown dry with nitrogen. The solvents in the remaining photoresist pattern are then completely removed by a 10 minute bake at 150°C.

When the complete pattern has been defined on both ends of the thermopile, the tungsten is etched in the following solution:

6 grams NaOH (sodium hydroxide)
12.6 grams $K_2Fe(CN)_6$ (potassium ferricyanide)
20 ml distilled H_2O

The thermopile is held with plastic coated tweezers and one end at a time is lowered into the solution for 45 seconds. The etching solution is kept at room temperature and stirred continually during the etching operation. Etching is stopped with a distilled water rinse followed by blowing dry with nitrogen. The etching progress is then checked with a microscope. This procedure is repeated until the tungsten is completely removed as evidenced by the metallic color of the SiGe. Care is taken to avoid overetching which will result in undesirable undercutting of the pattern.

FIGURE 8. Photoresist Spinning Apparatus

FIGURE 9. Thermopile Holding Fixture Used in the Spinning Operation

FIGURE 10. Mask Alignment and Exposure Apparatus

After etching both ends, the thermopile is rinsed in running distilled water followed by a forced spray rinse in distilled water and then blown dry with nitrogen.

Normally, as a last step in the photolithographic process, the photoresist is removed, however, for reasons explained later, the photoresist will be left on. The thermopiles are individually wrapped in lint free cloth and placed in plastic containers. A complete photolithographic procedure is detailed in Appendix D.

ELECTROLYTIC ETCH

Following the photolithographic process, the thermopiles are checked electrically for shorts between channels and total resistance of each channel and each contact are measured. All measurements are made using a four point probe technique except the interchannel resistance which is measured with an ohmmeter having a 16 megohm sensitivity. It is desirable that the interchannel resistance of a thermopile be greater than 16 megohms. Occasionally, sputtered tungsten will remain in voids in the glass separator between the two channels. The tungsten not removed from these voids by the standard photolithographic process will form electrical leakage paths and even cause direct shorts. An electrolytic etch process developed by F. G. Yost, 2431, is used to remove the tungsten from these voids.

The photoresist not removed from the thermopile following the photolithographic process will protect the contact areas from the electrolytic etch. The thermopile is immersed into an electrolytic bath consisting of 5% sodium hydroxide (by wt.) in deionized water. A positive lead from a 10 volt dc source is connected to both channels of the thermopile, and the negative lead is connected to a copper strip in the solution. The thermopile is kept in the solution for 15 seconds while the electrolyte is stirred gently to liberate bubbles which form on the surface of the thermopile. The thermopile is then rinsed in deionized water followed by a methyl alcohol rinse and air dry.

The thermopile is rechecked for shorts and the etch process repeated if necessary. Finally, the photoresist is removed by scrubbing the thermopile with a cotton bud and trichloroethylene.

The completed contacts on both ends of a thermopile is shown in Figure 11. Figure 12 shows the output connections to the thermopile made by eutectic bonding gold plated Kovar pads to the outermost elements using an AuSi preform alloy.¹ Gold ribbon leads (.005 cm x .038 cm) are attached to the pads by a parallel gap welding method.² The side contact pads and lead attachment process is conducted by Organization 7121-1 then returned to Organization 2431 for final cleaning.

FINAL CLEANING

Since the sputtering operation, the thermopiles have been subjected to a large amount of handling. Therefore, they undergo another cleaning using the same cleaning procedure as described before with the exception of the hydrofluoric etch step. Following the final cleaning, the thermopiles are again individually wrapped in clean lint free cloth and placed in plastic containers. This completes the intra-element contact processes of the thermopiles.

DISCUSSION

Metallization and photolithographic processes have been described for applying tungsten interconnections to the RTG thermopiles. The processes so described will produce tungsten films with good adhesion and low contact resistance providing the procedures are closely followed. During a six month period, 89 thermopiles were processed with an effective yield of 80%. Major yield losses were attributed to high contact resistance of the contacts from the second end metallization.

a

b

FIGURE 11. (a) The hot end of the thermopile showing 22 contacts per channel.
(b) The cold end of the thermopile showing 21 contacts per channel.

FIGURE 12. A Completed Thermopile With Output Connections

Discounting the physical dimensions of the thermopile, pattern definition is a straight-forward photolithographic process. Because of its unusual dimensions as opposed to a standard flat type substrate, special fixturing is required to handle the thermopile through the patterning processes. This special fixturing is necessary for both the photoresist application and for the alignment/exposure system. A method for masking or protecting one end of the thermopile while the opposite end is being exposed could perhaps be incorporated into the design of the special holding fixture for the alignment system.

Improved dimensional control of the glassing operation would simplify the mask alignment whereby only one or possibly two alignments and exposures would be necessary to pattern one end of the thermopile, since the transfer of this technology this has been accomplished.⁹

A device for holding the thermopile during etching should be designed in such a manner that would permit etching of either one or both ends of the thermopile at a time.

Contact resistance of the second end metallization has been noted to be consistently higher than that of the first end by a ratio of 1.5 to 2.5. Since the metallization process, as described, permits metallization of only one end at a time, the second end receives a double presputter conditioning. Consequently, the second end has a longer exposure to vacuum system ambients. An Auger analysis⁸ of experimental samples revealed that the second end metallization contained a greater oxide formation at the SiGe-W interface. It has also been observed that the ratio of hot end to cold end resistance is greater for higher values of substrate temperature during first end metallization.

Tungsten flake deposition on the surface of the thermopiles during sputtering is controlled to a limited extent by periodic cleaning of the sputtering furniture. The removal of tungsten deposits involves complete disassembly of the thermopile

fixture and the sputtering furniture. The tungsten deposits are then removed by an air blast hone using glass beam abrasive followed by a deionized water rinse. Prior to any subsequent thermopile metallization, the system is subjected to a 20 minute sputtering run to coat the newly cleaned surfaces with a thin film of tungsten to protect the thermopiles from stainless steel contamination.

APPENDIX A

PRESPUTTER CLEANING PROCEDURE

The following procedure is used to clean the thermopiles prior to the tungsten sputtering. The cleaning (Part B) is to be done less than 10 minutes prior to loading the piles into the sputtering apparatus.

A. Prescrub (for removal of gross contaminants)

1. Materials

- a. Shallow dish
 - b. Teflon coated tweezers - Bel-Arts Products, Applied Coating Division, Pequannock, New Jersey, Cat. #F-37935.
 - c. Cotton buds - Johnson & Johnson, New Brunswick, New Jersey.
 - d. Trichloroethylene - A. R. Stock #684728.
 - e. Methyl Alcohol - A. R. (ACS) stock #682190.
2. Scrub all surfaces of thermopiles in trichloroethylene using tweezers and a cotton bud.
 3. Scrub all surfaces of thermopiles in methyl alcohol using tweezers and a cotton bud.

B. Cleaning

1. Equipment Used

- a. Ultrasonic Cleaner
 - 1) Westinghouse Model #WEH-10
 - 2) Power density: 7.6 watts per square inch.

b. Fixturing

1) Holding fixtures

A-1401 Basket

A-01 Handle

Products of: Fluoroware, Inc.
Chaska, Minnesota

NOTE: Every second partition has been removed
from the basket.

c. Safety Equipment

1) Fume hood

2) Apron

3) Polyethylene gloves

d) Face shield

d. Timer

e. Chemicals

1) Trichloroethylene - A.R. stock #684728

2) D.I. water - 18 megohm

3) Isopropyl Alcohol - A.R. (ACS) stock #682182

4) Dry Nitrogen Gas, liquid pumped, stock #681422

5) HF Solution

a) 8 parts D.I. water

b) 1 part hydrofluoric acid H.E. (ACS) stock #687255

6) Detergent Solution

a) 20% (by volume) D.I. water

79.3% (by volume) Isopropyl alcohol-
A.R. (ACS) stock #682182

.5% (by volume) Span 80 - Product of:

Atlas Powder Co.
Wilmington, Delaware
Sandia Spec. 4702401

.2% (by volume) Remex 690 - Product of:

Atlas Powder Co.
Wilmington, Delaware
Sandia Spec. 4702403

2. Procedure

All of the following operations are to be performed under the fume hood. Use the timer for timed operations. Wear face shield, polyethylene gloves, and apron at all times while handling an open container of hydrofluoric acid.

- a. Place the thermopiles in holding fixture.
- b. Ultrasonically clean the thermopiles in trichloroethylene for two minutes and then blow dry with dry nitrogen.
- c. Ultrasonically clean the thermopiles in the detergent solution for two minutes. Shake off the excess liquid.
- d. Ultrasonically clean the thermopiles in D.I. water for two minutes. Blow off excess liquid with dry nitrogen.
- e. Etch the thermopiles in the HF solution for 20 seconds at room temperature.
- f. Ultrasonically clean the thermopiles in D.I. water for two minutes. Blow off the excess liquid with dry nitrogen.
- g. Ultrasonically clean the thermopiles in isopropyl alcohol for two minutes. Blow dry with nitrogen.
- h. Immediately load the thermopiles into the sputtering equipment and pump down the vacuum system.

APPENDIX B

THERMOPILE TUNGSTEN METALLIZATION PROCEDURE

GENERAL INSTRUCTIONS

During loading and unloading, the operator shall wear polyethylene gloves. Teflon coated tweezers should be used in handling the thermopiles. To avoid damage to the thermopiles, tweezers should be replaced frequently as the teflon coating wears off exposing the bare metal.

Each thermopile is identified by a number inscribed on its side. As the number is read, the lefthand end of the thermopile has been designated as the "cold end." The righthand end of the thermopile is the "hot end." For the first end metallization, the thermopiles are loaded into the sputtering fixture with the cold end facing upward. Care must be exercised in handling the thermopiles to avoid damage. They should be lowered into the fixture rather than dropped. The thermopile should be gripped with the tweezers near the center, well away from the edges, to avoid chipping. At no time during loading should the ends of the thermopiles come in contact with any object or substance.

The thermopiles rest on glass pads placed on top of stainless steel blocks. Machine screws at the bottom of the sputtering fixture are for the purpose of adjusting the height of each thermopile so that all thermopiles are flush with the top surface of the fixture.

Two glass control samples are included in each deposition run for the purpose of film thickness and film resistivity evaluation.

The pads and control samples are Corning 7059 glass. They receive the same cleaning procedure as the thermopiles with the exception of the HF etch step.

All vacant positions in the fixture are filled with stainless steel blocks. A thin film of tungsten is sputtered on the top surface of each block to prevent stainless steel contamination of the thermopiles.

Periodic cleaning of the sputtering furniture is required to remove the buildup of tungsten on the thermopile fixture and the surrounding furniture to prevent flake deposits on the thermopiles. The tungsten deposit is removed by an air blast hone with a glass bead abrasive followed by a deionized water rinse to remove the residual abrasive material. The operation involves a complete disassembly of the thermopile fixture and the sputtering furniture.

The pneumatically operated foreline valve requires a regulated air pressure of 20 psi. The argon pressure regulator should be set for a static pressure of 8 psi.

I. LOADING (FIRST DAY)

1. Lower thermopiles into the sputtering fixture.
2. Adjust the height of each thermopile flush with the top surface of the fixture by means of the adjusting screws at the bottom of the fixture. Secure the screws in position by tightening the locknut. The screw adjustments are made with special tools provided.
3. Rotate the loaded fixture under the shutter. An indexing mark on the rotating platform holding the thermopile fixture will be coincident with the vertical shield plate separating the two target areas. A second mark on the platform will appear at the

same position when the thermopile fixture is directly under the target. These markings are useful for accurate positioning of the thermopile fixture when viewed through the bell jar window.

4. Open the throttle valve for maximum throughput. Secure in position with the knurled brass nut.
5. Lower the bell jar onto the base plate and position the viewing port so that the indexing mark on the rotating platform and the vertical shield plate are clearly visible.

II. PUMPDOWN (FIRST DAY)

1. Turn on the main power to the console by pressing the button marked "POWER."
2. Turn on the power switch on the Veeco RG-2A Vacuum Gauge. Make sure the DEGAS switch is in the down position (OFF) before turning on the power.
3. Turn on the Thermistor Gauge GT-340 by switching to position BELLJAR HIGH STA 2. After 5 minutes warm-up, the meter should indicate in the black region on the scale and the GT-005 Controller red light should be on in the N.O. circuit position.
4. Press the START button. This will start the rough pump and open the foreline valve. The turbomolecular pump will start when the belljar is rough pumped to approximately 1 Torr. The red light on the GT-005 Controller will switch from N.O. circuit to N.C. circuit.
5. Set the PRESSURE MULTIPLIER switch to the 10^{-4} position (temporarily ignore the red flashing warning light) and turn on the filament switch when the pressure indicated on the GT-340 Thermistor Gauge is less than 1 millitorr. Switch the PRESSURE MULTIPLIER to the 10^{-5} Torr position as soon as the belljar pressure reaches 1×10^{-4} Torr.

The red warning light will stop flashing. The purpose of the warning light is to alert the operator that the pressure selector switch must be in the 10^{-5} Torr position during the belljar bakeout period.

III. BAKEOUT

1. Turn on the power to the belljar heater and substrate heater when the belljar pressure is 2×10^{-5} Torr or less.
2. Set the belljar bakeout timer for an 8 hour bake.
3. Set the power to the substrate heater at 24 Vdc and 4.4A.

IV. SPUTTERING (SECOND DAY)

1. Turn on the Hastings Mass Flowmeter.
2. Turn on the GP-310 Pirani Gauge. Set the selector switch to the 100 position.
3. Set the selector switch on the Thermistor Gauge to position FORELINE HIGH STA 1.
4. Turn on the Inert Gas Purifier. Temperature control should be set to the pencil mark on the scale. This setting will bring the purifier oven temperature to 900°C. A slight temperature overshoot will occur on initial warm-up. Do not try to adjust for the overshoot.
5. Switch GT-340 Thermistor Gauge from BELLJAR position to FORELINE STA 1 HIGH position.
6. Turn on the Doric Temperature Indicator and record the temperature.
7. Record the ultimate belljar pressure as indicated on the Veeco RG-2A Vacuum Gauge.
8. Turn off the power switch on the Veeco RG-2A Vacuum Gauge.
9. Open both toggle valves on the Inert Gas Purifier.

10. Open the argon leak valve to approximately 92 on the indicator.
Make final adjustments to maintain a gas flow of 145 SCCM as indicated on the Hastings Mass Flowmeter.
11. Adjust the throttle valve to indicate 20 millitorr on the Pirani Gauge* The foreline pressure should be approximately 190 millitorr (GT-340 Thermistor Gauge).
12. Set the voltage control of the SDC-100 high voltage dc power supply to 0 and turn on the power switch. Press the high voltage button and adjust the voltage control to indicate 50 ma on the milliammeter. The voltmeter should indicate about 3800 volts.
13. Presputter for 30 minutes (substrates under shutter) making periodic adjustments of the high voltage control to maintain 50 milliamperes of current until the voltage reaches 4500 volts.
14. Record the substrate temperature.
15. Begin to sputter onto the thermopiles by rotating the platform under the target. The thermopiles will be in position when the second indexing mark is in alignment with the vertical shield plate.
16. Sputter for three hours and record every 30 minutes the following:
 - 16.1 Sputtering Voltage. Make adjustments if necessary to maintain 4500 volts.
 - 16.2 Sputtering Current.
 - 16.3 Argon Flow. Make necessary adjustments to maintain 145 SCCM.
 - 16.4 Belljar Pressure.
 - 16.5 Foreline Pressure.
 - 16.6 Substrate Temperature.

*The Pirani Gauge is calibrated for air. Argon pressure at this setting is 31 millitorr.

17. At the end of the three hour sputtering run, turn off the high voltage power supply by returning the voltage control to 0 and turning off the power switch.
18. Turn off the substrate heater power supply.
19. Close the argon leak valve to 5 on the indicator.
20. Open the throttle valve for maximum throughput.
21. Turn off the power switches on the Hastings Flowmeter and the GP-310 Pirani Gauge.
22. Turn off the power switch and close the toggle valves on the Inert Gas Purifier.

V. SHUTDOWN

1. Allow the substrate temperature to drop to 150°C before proceeding to shutting down the system.
2. Press the STOP button. This switch will turn off the rough pump and turbo pump, close the foreline valve, and open the vent valve to backfill the belljar with argon to near atmospheric pressure.
3. Turn off the main power by pressing the button marked POWER.
4. Allow the substrate temperature to reach room ambient before opening the belljar. This will normally take an overnight cooling period.

VI. SECOND END METALLIZATION (THIRD & FOURTH DAY)

1. Remove the thermopiles from the fixture by inserting a copper wire probe into the hole of the adjusting screw, lifting the thermopile high enough to grasp with the teflon coated tweezers. Reverse the position of each thermopile one at a time. Replace the control sample with new substrates.
2. Repeat steps I through V for the second end metallization.

VII. FIFTH DAY

1. Remove the thermopiles from the sputtering fixture and wrap each thermopile individually in a piece of cloth or similar material and place them into plastic containers for storage and safe transport.

APPENDIX C

KMER PHOTORESIST FILTERING PROCEDURE

Filtration of KMER[®] photoresist shall be done in a laminar flow clean fume hood. First, fill a 170mm x 90mm crystallizing dish 1/4 full with trichloroethylene and set dish aside in the hood. Clean a 3 ounce brown medicine dropper bottle (with glass eyedropper) with methylene chloride by swabbing the eyedropper (inside and out) with a cotton swab and by pouring a small quantity of the methylene chloride into the bottle, shaking and then pouring out the solution. Repeat this cleaning procedure three times. Holding the bottle and cap with eyedropper in one hand, blow dry with nitrogen using the Paasche air spray gun. Set the bottle assembly aside on a filter paper in the fume hood.

Assemble the high pressure stainless steel filter apparatus according to Millipore[®] instructions making sure the 14 μ Duralon filter is positioned such that the grid pattern side of the filter will be upstream of the photoresist flow. Assemble and clamp the filter apparatus (except for the top cap) to the laboratory ring stand. Set the assembly to one side in the fume hood. Pour 15 ml of KMER thinner into a 50 ml graduate. Remove the KMER photoresist from the refrigerator and add 30 ml of photoresist to the thinner solution.

[®] Trademark, Eastman Kodak Co., Rochester, N.Y., "Kodak Metal Etch Resist."

[®] Trademark, Millipore Corp., Bedford, Mass., Catalog No. NCWG04700.

Stir with a teflon coated stirring rod and pour the solution into the filter assembly. Screw on the cap assembly and attach the length of tygon tubing to a nitrogen tank. Place the clean medicine dropper bottle under the filter spout and slowly turn the throttle on the nitrogen tank allowing two or three pounds of pressure into the filter housing, forcing the photoresist through the 14 μ filter. Increase pressure as needed (approximately 8 psi) to attain a flow of about 1/16 inch in diameter.

When the last of the photoresist is filtered, turn the nitrogen throttle to the left to shut off the flow of nitrogen and allow the line to bleed. Remove the medicine dropper bottle (with photoresist) from the stand and cap immediately with cap and eyedropper. Place the bottle in its original box and label both bottle and box with the following information:

KMER 2:1
14 μ Filter
(Date)

The filtering is now completed and only the clean up remains. This must be done very thoroughly so that all equipment and apparatus parts will be clean for the next filtering. The surfaces must be free of any traces of old photoresist. Remove the cap and tubing from the filter housing and return to storage. Disassemble the filter housing and discard the 14 μ filter. Place the parts in the crystallizing dish of trichloroethylene and agitate in the solution. Remove the parts and place them on a clean disposable paper towel. Replace the dirty trichloroethylene with the same quantity of a fresh supply. Again place parts in the fresh solution and agitate. Remove and thoroughly dry parts with lint free cloth. Clean the glass graduate and stirring rod in the same manner through two solutions of trichloroethylene. Discard all trichloroethylene and rinse the crystallizing dish with fresh solution, empty and dry. Return filter parts to container and to storage.

APPENDIX D

PHOTOLITHOGRAPHY PROCEDURE

Due to the very brittle and fragile nature of the silicon-germanium elements, thermopiles must be handled very carefully at all times. Photolithographic procedures on these thermopiles shall be done in a clean room (class 10,000 or better) illuminated with safe yellow lights and have available a laminar flow clean fume hood. Operators shall wear clean lab coats and keep shoes relatively clean by using either tacky floor mats placed at entryway or utilize a shoe cleaning machine. Extra precautions should be observed from the time photoresist is applied to the thermopile through the developing operation. During this particular part of the photolithographic process the photoresist must be protected by keeping thermopiles in covered petrie dishes and maintaining subsequent work areas in clean benches or hoods. Teflon tweezers shall be used when handling thermopiles and be replaced with new tweezers when the teflon coating wears off exposing the bare metal. Operators shall wear finger cots or polyethylene gloves when handling the thermopiles.

Each thermopile is identified by a number inscribed on its side. As the number is read, the lefthand end of the thermopile has been designated as the "cold end" requiring 21 tungsten contact strips to be defined. The righthand end of the thermopile is the "hot end" and requires that 22 tungsten contact strips be defined.

Since dimensional control of the SiGe elements and the glassing between elements has not been accurate enough to allow for a single exposure of a complete contact pattern, it is necessary to make several exposures on each channel. The number of exposures necessary for each channel is determined by the accumulation of errors in the size of both the SiGe elements and the glass areas and the size of the contact pad required.

Three sets of masks have been made that allow 1, 3, 4, 5, 6, 8, 12, 21, 22 contact strips to be exposed at one time on the channel and provide for three different sizes of contact strips, each set increasing in size by 1 mil. (See Figure 7). The smallest pad size allows for a 3 mil border to extend from the contact strip to the edge of the elements being connected. The other two contact strip sizes increase by 1 mil each, thus giving a 2 mil border and a 1 mil border around the tungsten contact strip.

By using the medium, or 2 mil size, contact strip dimension and exposing 4 contact strips at one time, six exposures are required for each channel on both the hot end and the cold end making a total of 24 exposures necessary to complete one thermopile. This is the size strip and number of exposures presently being used. Procedure for photolithographic pattern definition is as follows:

1. Coat appropriate surface with KMER photoresist. The photoresist used is the negative acting KMER (diluted 2 parts KMER[®] and 1 part KMER thinner) and filtered through a Millipore[®] 14 μ filter. The resist filtering

[®] Trademark, Eastman Kodak Co., Rochester, N.Y., "Kodak Metal Etch Resist."

[®] Trademark, Millipore Corp., Bedford, Mass., Catalog No. NCWG04700.

procedure is included in Appendix C. Position the spinner fixture (Figure 9) on the photoresist spinner shaft and finger press for friction hold. Using teflon coated tweezers, the thermopile is grasped toward one end and placed in the spinner fixture so that approximately $1/8$ " of the thermopile remains above the fixture. The thermopile is held in this position by tightening the set screw located on the side of the fixture. After securing the thermopile in spinner fixture a small amount of filtered photoresist is placed on the end of the thermopile with a glass eye dropper. The photoresist should completely cover the end of the thermopile. Set the Variac dial on front of spinner at 3.2. This setting will result in a spin speed of 3000 rpm.

2. Spin at 3000 rpm for 30 seconds. Turn switch to ON position and spin the thermopile with the photoresist at 3000 rpm for 30 seconds. At the end of 30 seconds, turn the switch to the OFF position. The end of the thermopile is again held with tweezers and the side set screw is loosened to release the thermopile from the fixture. Using caution, the thermopile is turned end for end and now the resist coated end is lowered into the fixture and held in place by tightening the side set screw. Again, allow approximately $1/8$ " of the thermopile to remain above the surface of the fixture. Do not allow the thermopile to drop into the fixture as chipping of the SiGe may occur and/or scratching of the photoresist. Again, place a small amount of photoresist on the end of the thermopile and repeat the spinning operation as mentioned above. Again, holding the thermopile with tweezers, release the side set screw, lift thermopile from fixture and carefully place in a glass petrie dish. Place cover over dish.

3. Pre-bake at 110°C for 10 minutes. Place petrie dish with thermopile into 110°C oven, slide the petrie dish cover to one side, close the door and set timer for 10 minutes. At the end of 10 minutes, open oven door and slide the cover over the dish and remove from oven to a clean area and allow petrie dish and thermopile to cool to room temperature.
4. Expose through appropriate mask for 30 seconds. Place mask in the mask holder on the alignment machine and secure by sliding lock bar (located on mask holder) into perpendicular position. Make sure the emulsion side of the mask is facing down, this is important. Leave mask holder in the upright position. Hold the resist coated thermopile with tweezers and insert the "cold end" into the holding fixture. The holding fixture is held in a horizontal position in the lefthand of the operator. Slide the thermopile into the fixture until only approximately 1/32" of the "hot end" remains above the surface of the fixture. Secure the thermopile in this position by tightening the set screw located on the side of the fixture. Still holding the fixture in the lefthand, place a pre-cut piece of black paper (cut to the size of the end of the thermopile) over the "cold end" of the thermopile and secure with 1/8" wide pieces of masking tape. It is necessary to protect the photoresist on this end of the thermopile from the light while exposures are made on the "hot end." Place the holding fixture in the recessed area on vertical lift of the alignment machine. The holding fixture rests in this recessed area and requires no further means of fastening or securing in place. Lower the mask and mask holder and secure by sliding lock bar into perpendicular position. Thermopile and mask are now in position to start the aligning procedure. In aligning any substrate and mask in this machine, five control knobs are used. Two micrometer type knobs are positioned at opposite sides of

the stage where the mask holder is located and control movement of the stage (and mask) in the X and Y directions. This could be indicated as left to right and front to back direction. A third knob located on the right front side of the machine just below the stage controls the rotational movement of the stage and mask. The remaining two knobs located at the bottom front of the machine controls the vertical movement of the substrate or thermopile. These five knobs will control all movements of both mask and thermopile to achieve the correct alignment required for the exposure. To simplify, the stage holding the mask is above the thermopile and can move in three directions: left to right, front to back, and rotate in an arc. The thermopile is positioned underneath the mask and when the correct alignment has been made, will rise vertically and make contact with the mask, thus, the term contact printing. Located back of the stage on this machine is the housing holding the microscope and the exposure lamp. This entire housing will slide back and forth behind the stage moving either the microscope into position above the mask and stage or move the exposure lamp into position above the mask and stage. It is necessary for the operator to position the microscope above the mask and view both the thermopile and the mask while the aligning process is performed. When the thermopile and mask are in alignment with each other, contact is made between the two and all is ready for the exposure. Slide the housing and microscope to the left. This will bring the exposure lamp into position. Set the timer located in the same housing to 30 seconds. Switch the exposure light switch to START and allow the timer to time the exposure and to automatically shut off the exposure lamp at the end of the 30 seconds.

Immediately after the exposure has been made separate the mask and thermopile by lowering the vertical lift using the two knobs at lower front of the machine. It is important that this separation be made before continuing with any succeeding steps.

5. Repeat Step 4 as many times as required. After separation of mask and thermopile, the operator must now move the mask eight elements down the thermopile channel and realign the mask to these next eight elements. The alignment is done in the same manner as the previous alignment in Step 4. As each group of eight elements are exposed, continue moving to the next group until the entire channel has been exposed. At this point, remove the mask from the mask holder, rotate it 180° and replace it in the mask holder and secure. This rotation of the mask is necessary because of the off center location of the pattern in the overall 2" x 2" size of the glass. By doing this, the operator will experience no trouble later on in running out of travel space. Again position the mask over the second channel and proceed with the same type of alignments and exposures as that done on the first channel. After completing the "hot end" of the thermopile, separate mask and thermopile, lift mask holder into upright position and remove holding fixture (with thermopile) from the machine. Holding the fixture in the lefthand, remove the protective black paper cover from the "cold end" and slide the thermopile out of the fixture. Reverse the thermopile and slide the exposed "hot end" into the fixture until only about 1/32" of the "cold end" remains above the surface of the fixture. Secure the thermopile in place by tightening set screw at side of fixture. Place the protective black cover on the "hot end" and position the fixture back onto the vertical life mechanism. Rotate the mask 180° to return it to the original position and continue the same aligning steps as listed in Step 4. Finish exposing both channels

on the "cold end." Separate the mask and thermopile, raise the mask holder into the upright position and remove the holding fixture from the machine. Remove the thermopile from the fixture by loosening the set screw at the side of the fixture and remove the protective black cover from the "hot end." Place the thermopile in petrie dish and cover.

6. Develop using spray KMER developer for approximately 25-30 seconds.

Transfer the petrie dish with the thermopile to a clean hood area.

Developing KMER photoresist is done by spraying (Paasche Air Spray) with KMER developer solution for approximately 25-30 seconds. Using teflon tweezers, the thermopile is held by the middle section and KMER developer sprayed is over the resist area (both ends) for approximately 25 seconds per end.

The thermopile is rotated back and forth so the spray reaches both ends and developing is completed on both ends at the same time. LEAVE

THERMOPILE WET WITH DEVELOPER SOLUTION.

7. Spray with isopropyl alcohol and blow dry. . Set spray bottle aside and

pick up a second spray bottle which contains isopropyl alcohol and spray rinse the thermopile from end to end. Spray rinse for a total of approximately 25 seconds and blow dry with nitrogen air from the same spray gun. This is done by pushing the button on the spray gun down and forward. To spray with a solution, the button on the spray gun is pushed down and back. Return thermopile to the petrie dish and transfer to the microscope area. The pattern definition should be microscopically inspected at this point to determine that alignment is correct and the proper amount of developing was achieved. After making sure pattern definition is adequate, return the thermopile to the covered petrie dish.

8. Post-bake at 150°C for 10 minutes. Check the temperature of the post-bake oven and place the petrie dish inside. Slide the petrie dish cover to one side and close the oven door. Set the timer for 10 minutes.
9. Store in clean, dust free area. At the end of the post-bake period, cover the petrie dish and remove from oven. Store the covered dish with thermopile in a clean, light safe, dust free area.
10. Prepare tungsten etch. The formula for tungsten etch is as follows:

6 grams NaOH (sodium hydroxide)
12.6 grams $K_3Fe(CN)_6$ (potassium ferricyanide)
200 ml distilled H_2O

Prepare this solution by first placing 200 ml of distilled water in a 250 ml beaker. Using a balance, weigh the sodium hydroxide and add to the water and stir until dissolved. Next, weigh the potassium ferricyanide, add to the solution and stir until dissolved. Pour and store this solution in a flask which has a ground glass stopper. This will prevent evaporation of the solution.

11. Etch the tungsten film. Place a 1 inch long teflon coated magnet into a 250 ml size beaker and pour in approximately 200 ml of the etch solution. Put the beaker on a magnetic stirrer and turn the power switch to ON position to activate the magnetic field which causes the magnet to spin and thus provides a constant stirring of the etch solution. Using teflon coated tweezers grasp the thermopile by the end and lower the opposite end into the etch solution (immerse approximately one-half the thermopile) at the same time activate a timer clock. Hold the thermopile in this position in the stirring solution for approximately 45 seconds. At this point, remove the thermopile from the solution, stopping the timer clock at the same time and hold the thermopile in running distilled H_2O for 20-30 seconds. Using the Paasche Air Spray equipment, spray the thermopile with

distilled water at a pressure of 40 psi for 20 seconds and blow dry with nitrogen. Using a microscope, inspect the etched end of the thermopile. The tungsten film will turn a dull jet black color as it is etching and after the tungsten is completely removed, the shiny metallic color of the polished SiGe will be visible. If the tungsten film is still black return the thermopile to the etchant and continue etching for approximately 20-30 seconds longer. Remove from the solution, rinse as previously explained and blow dry. Inspect again using a microscope. Repeat this etching step and inspection until the tungsten film is completely removed. It is desirable to etch only long enough to remove the tungsten film, otherwise continued etching will result in undercutting. When one end of the thermopile has been etched, reverse the thermopile and immerse the opposite end into the etchant. Repeat the process as described previously. After all etching is complete, thoroughly rinse the thermopile first in running distilled water, then forced spray rinsed in distilled water and blow dry with nitrogen. DO NOT REMOVE THE PHOTORESIST. Wrap the thermopile with lint free Texwipe cloth and place in covered plastic box.

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