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FABRICATION OF THIN FILM HEAT FLUX SENSORS

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ABSTRACT

Prototype thin film heat flux sensors have been constructed and tested. The sensors can be applied to propulsion system materials and components. The sensors can provide steady state and fast transient heat flux information. Fabrication of the sensor does not require any machining of the mounting surface. Heat flux is proportional to the temperature difference across the upper and lower surfaces of an insulation material. The sensor consists of an array of thermocouples on the upper and lower surfaces of a thin insulating layer. The thermocouples for the sensor are connected in a thermopile arrangement. A 100 thermocouple pair heat flux sensor has been fabricated on silicon wafers. The sensor produced an output voltage of 200-400 microvolts when exposed to a hot air heat gun. A 20 element thermocouple pair heat flux sensor has been fabricated on aluminum oxide sheet. Thermocouples are Pt-Pt/Rh with silicon dioxide as the insulating material. This sensor produced an output of 28 microvolts when exposed to the radiation of a furnace operating at 1000°C. Work is also underway to put this type of heat flux sensor on metal surfaces.

INTRODUCTION

The objective of this work is to research and develop thin film heat flux sensors for application on propulsion system materials and components used in space propulsion system environments. The design and development of space propulsion systems requires an accurate knowledge of the heat loading on all critical propulsion system components. The space shuttle propulsion system makes use of a very reactive fuel and oxidizer system (liquid hydrogen and oxygen). When ignited the components of the engine are highly stressed both mechanically and thermally. The space shuttle main engine high pressure fuel and oxidizer pumps undergo huge temperature variations. The temperatures vary from cryogenic to around 1000°C during start up. This temperature increase occurs in about 1/2 second and results in a very large heat flux. When the engine shuts down a very large reverse heat flux also occurs. The result is cracking of the components and spalling of coatings. Heat flux sensors can be used to monitor the condition of

critical components of an engine during operation. For example, a crack in a blade may cause a drastic change in the heat flux passing through the blade.

Thin film heat flux sensors can be especially valuable for propulsion system components since they can provide heat loading information with minimal perturbation of gas flows. Thin film sensors can provide steady state and fast transient heat flux information. Fabrication of the sensor does not require any machining of the mounting surface.

The approach is to pattern a layer of thin film thermocouples on a substrate, cover it with a thin film insulator, and then pattern another layer of thermocouples on top of the insulator. The two layers of thin film thermocouples are connected together in a thermopile arrangement. This provides a direct measurement of the heat flux by measuring the temperature difference across the insulator. This approach is similar to work done by others[1].

THEORY

Heat flux sensors determine heat flux by measuring the temperature difference across a sheet of thermally insulating material. The heat flow per unit time per unit area through an insulating material is expressed as

$$ Q = \frac{K(T_1 - T_2)}{t} $$

where K is the coefficient of thermal conductivity, t is the thickness, T_1 is the temperature of one face, and T_2 is the temperature of the other face. This equation demonstrates that as the insulating layer is made thicker, the temperature drop becomes larger for a constant heat flux. The larger the temperature drop, the easier it is to measure the temperature difference across the layer.

Most heat flux sensors make use of a single temperature sensor on the upper surface of an insulating layer and a single sensor on the lower surface. The insulating layer is at least several hundred micrometers thick for commercial sensors. The temperature sensor is usually a resistance thermometer or a thermocouple. A schematic diagram of a single thermocouple pair heat flux sensors is shown in figure 1a. The thermocouples are connected in series with similar metal leads connected together. The voltage produced across a pair of thermocouples is proportional to the temperature difference.

Heat flux that is encountered in an engine environment can vary from 1 kw/m² to 20 Mw/m². For a 10 micrometer thick silicon dioxide insulating layer a temperature difference of 5.2×10^{-3} K will be obtained for a heat flux of 1 kw/m². This small temperature difference is very

(a) SINGLE THERMOCOUPLE PAIR TEMPERATURE SENSOR.
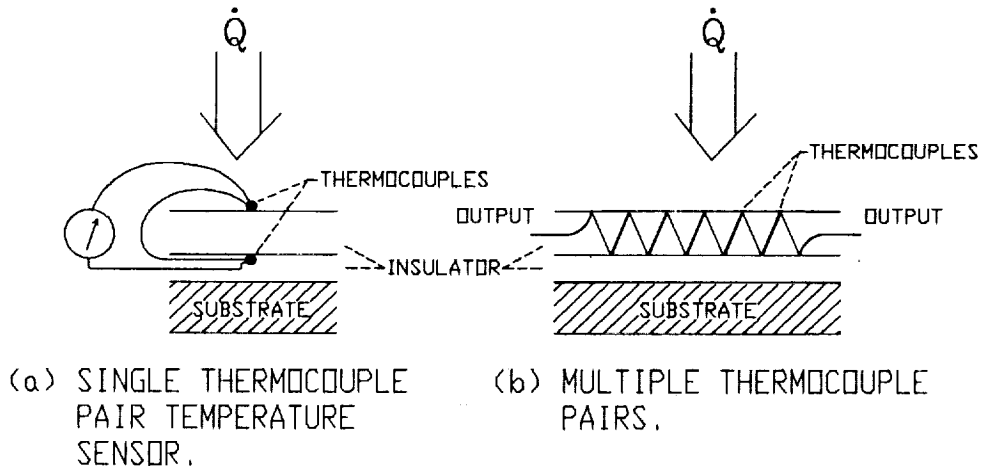
(b) MULTIPLE THERMOCOUPLE PAIRS.

Figure 1. Schematic diagram of thin film heat flux gage.

difficult to measure accurately with current temperature sensors. Note that a single thermocouple will generate a microvolt or less for this temperature difference. Typically sensors are constructed with an insulator thick enough to provide a temperature difference that can be easily measured.

SENSOR DESIGN

The heat flux sensor described here is more sensitive to the temperature difference across an insulating layer. This is done by patterning multiple temperature sensors on the upper and lower surfaces of an insulating layer in a thermopile arrangement.

A schematic diagram of a heat flux sensor with multiple thermocouple pairs connected in series is shown in figure 1b. The number of thermocouple pairs is limited by available space and resolution. The thermocouples are constructed of thin film metals sputter deposited on the upper and lower surfaces of the insulating layer. Upper and lower thermocouple pairs are electrically connected through the insulating layer. The thermocouples are connected so the voltages generated by the pairs add. The resultant output is a voltage signal equal to the number of thermocouple pairs times the voltage output of a single pair.

A three dimensional drawing of a ten thermocouple pair heat flux sensor is shown in figure 2. All the layers are deposited using RF diode sputtering. The thermocouple pairs are sputter deposited onto the insulator surface on the circumference of a small circle. This configuration was chosen so that the sensor measures only the heat flux normal to the surface. The connecting wires are sputter deposited through holes in the insulator on the circumference of a larger circle. This is done so the connecting wires do not disturb the heat flow in the vicinity of the thermocouple junctions.
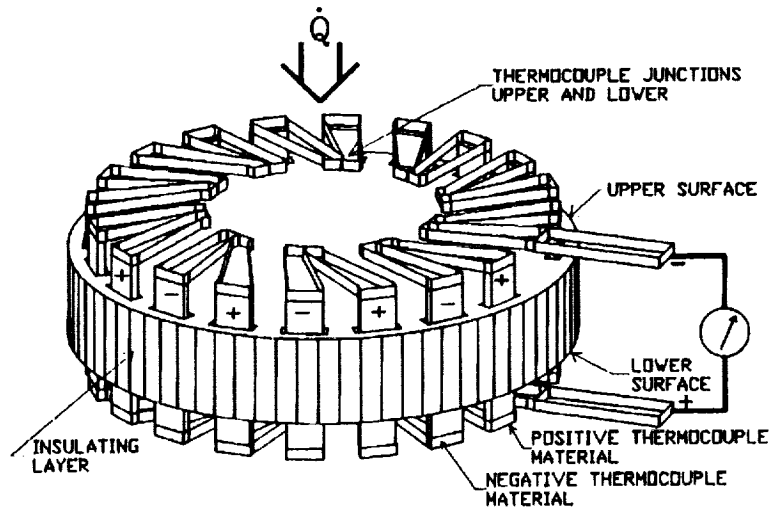
Figure 2. Three dimensional drawing of thin film heat flux gage. A gage with 10 thermocouple pairs is shown.

SENSOR FABRICATION

This high output thin film heat flux sensor can be applied directly to the surface of engine parts. An insulating layer must be deposited first if the surface is a metal.

In order to fabricate a thermocouple two different types of metals must be patterned onto the surface of the substrate. They must overlap one another in only one small area to produce a thermocouple junction. The thermocouples are patterned using a photoresist lift-off technique. Figure 3 displays the steps that are required for the lift-off technique. The substrate is initially coated with an insulating layer (figure 3a). Positive photoresist is applied to the surface and soft baked (figure 3b). The photoresist is then exposed with UV light through a photomask (figure 3c). The photoresist is then processed in a developer to remove the exposed resist. The resist is then dried and hard baked (figure 3d). The next step is to sputter deposit the first metal onto the surface of the sample (figure 3e). This results in the metal covering everything including the areas where the photoresist was developed away. The final step of the lift-off technique is to remove all the photoresist (figure 3f). This also removes the sputtered metal on top of the resist but not in the areas where there was no resist. The result is a metal pattern, on the sample, that is a replica of the photomask.

The process of fabricating a complete heat flux sensor consists of five steps. These five steps are shown in figure 4. The substrate is initially coated with a sputter deposited insulating layer of aluminum oxide. The first step uses the lift-off technique to pattern the first thermocouple layer. The second step also uses the lift-off technique to pattern the second thermocouple layer.
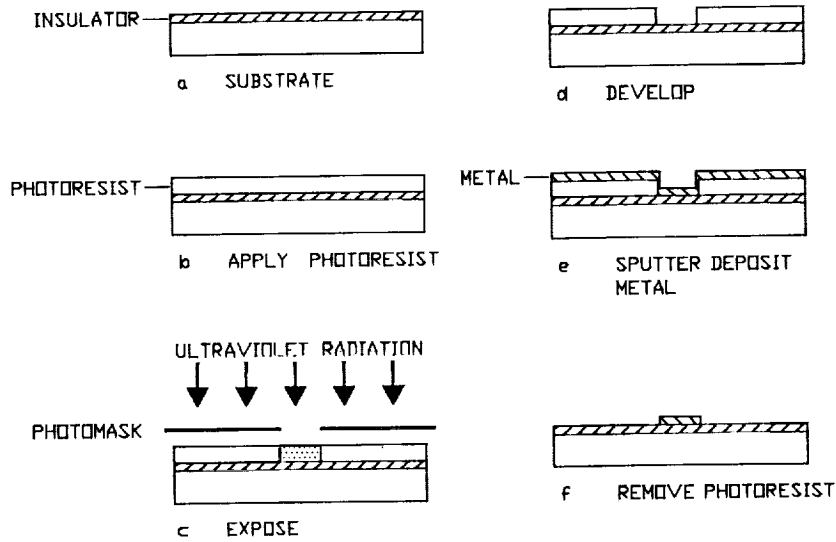
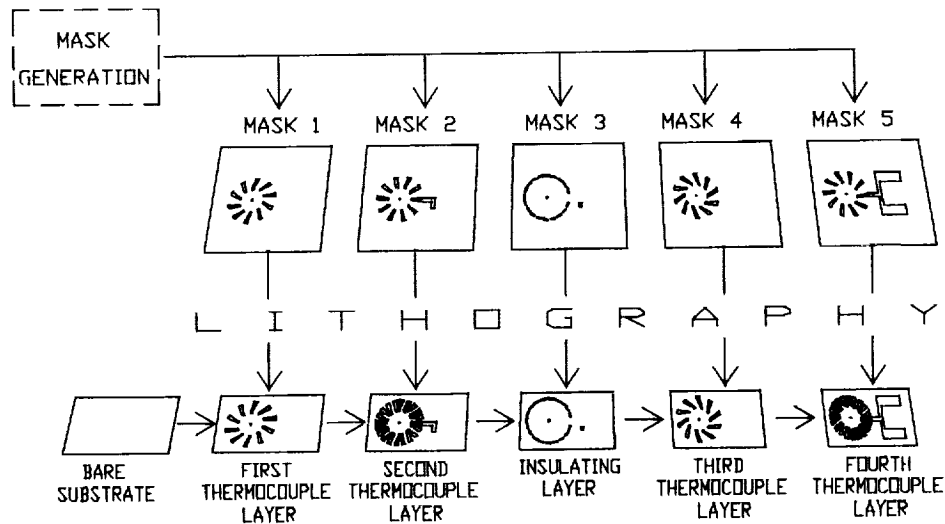**Figure 3. Diagram of thin film heat flux gage lift-off process.**

Figure 4. Processing steps used to fabricate thin film heat flux gage.

The third step consists of sputtering an insulator (such as SiO_2) over the lower thermocouple layer. The substrate is then coated with photoresist again and exposed with the mask shown in figure 4 (mask 3). The photoresist developer opens holes in the photoresist to expose the insulator for etching. A suitable etchant (buffered HF for SiO_2) is used to etch through the insulator to the underlying thermocouple layer. The final two steps use the lift-off technique again to pattern the upper two thermocouple layers. The result is a completed heat flux sensor with the upper and lower thermocouple layers connected in series through the holes in the insulator.
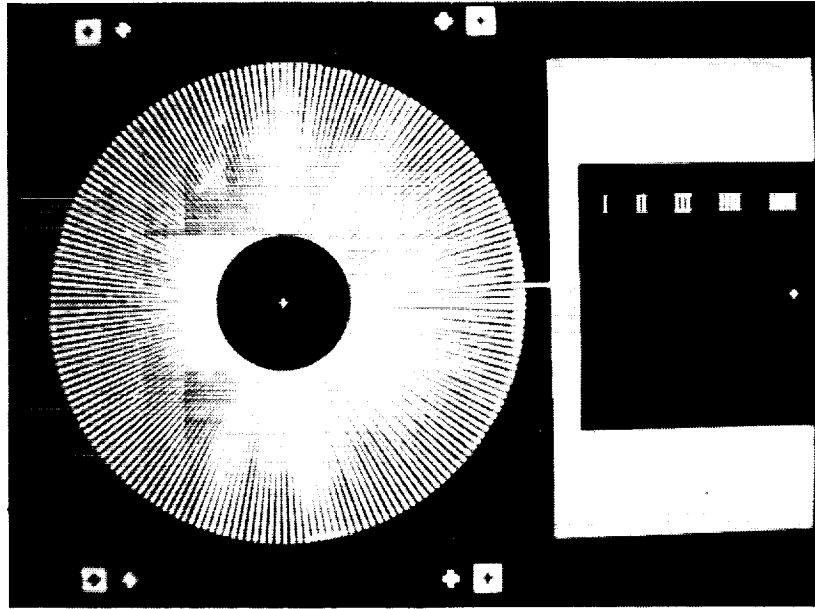
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Figure 5. Photo of complete 100 element thin film heat flux gage.

RESULTS

A thin film heat flux sensor fabricated on a silicon substrate is shown in figure 5. This sensor is a 100 thermocouple pair heat flux sensor. The thermocouple materials are chromel and alumel. The thermal insulating layer is silicon dioxide. Although the sensor is not without problems, it did work when tested. The resistance of the sensor was 171 ohms across the terminals. The resistance between sensor and substrate was greater than 10 megohms indicating no shorting through the insulator. The measured output using a heat gun as a source of heat was 0.2 to 0.4 millivolts. This heat flux sensor failed after repeated temperature cycling. The failure was an open circuit in at least one of the thermocouple legs. It appears that the failure was at the plated through holes of the thermal barrier layer. This is probably due to damage to the thermocouple metals by the buffered hydrofluoric acid used to etch the holes in the insulator.

In order to correct the open circuit problem it was decided to change the thermocouple materials to platinum and platinum/rhodium. Also it was felt that the current silicon substrate would not survive the high temperatures encountered during testing. As a result, the substrate was changed to aluminum oxide sheet. The pattern for the heat flux sensor was also changed from 100 thermocouple pairs to 20 thermocouple pairs. This was done to make the sensor easier to fabricate on the slightly rougher surface of aluminum oxide.

A 20 element heat flux sensor has been fabricated on a ceramic substrate (aluminum oxide) using Pt and Pt/Rh as thermocouple materials (see figure 6). The thermal insulating layer was 7 microns of silicon dioxide. The thickness of the thin film thermocouples was in the range 0.4 to 0.8 microns. The resistance of the sensor was 1000 ohms across the terminals. The previous
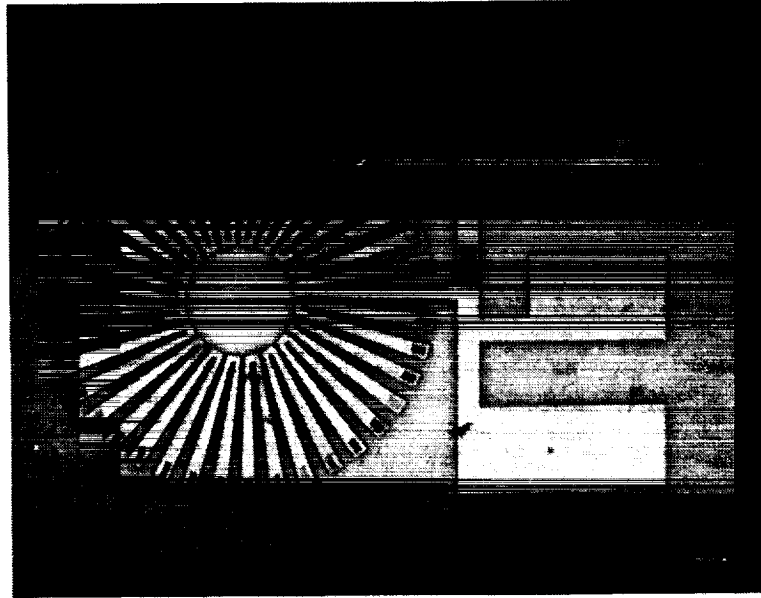
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Figure 6. Photo of complete 20 element thin film heat flux gage.

sensors, fabricated on silicon with chromel/alumel thermocouples, failed after one or more temperature cycles. This 20 element heat flux sensor continued to operate after repeated temperature cycling. The calculated output voltage for this sensor as a function of heat flux for two emissivities (1.0 & 0.5) is shown in figure 7. The heat flux sensor was tested by exposing the sensor to the radiation from a furnace operating at 775° C and at 900° C. Black body calculations, using Stefan's law, provided an estimate of the heat flux at each temperature. The output voltage for the sensor as a function of calculated heat flux is shown in figure 7 as two measured data points.

This sensor was also tested in a heat flux calibrator facility using an arc lamp. The sensor had no high emissivity coating since no insulating layer had been put over the top thermocouple layer. The sensor was tested up to a heat flux of 1.5 Mw/m^2. At this time the sensor broke in half. The voltage output of the sensor was also the wrong polarity indicating reverse heating of the sensor. It is believed the energy of the lamp was absorbed within the aluminum oxide substrate causing non-uniform heating and resulting in the breakage. In the future these sensors will be overcoated with an insulating layer of aluminum oxide followed by a coating of high temperature black paint. The paint will ensure that only the top surface of the sensor is heated.

There is also an additional problem with the use of aluminum oxide as a substrate material. It was found that when photoresist is used on polycrystalline aluminum oxide the exposure time is very critical. The light used to expose the photoresist is reflected off the polycrystalline grains resulting in extreme over exposure of the pattern from underneath. This makes it very difficult to

produce a good photoresist pattern. We have found that this problem can be eliminated by first sputtering 100-200 angstroms of aluminum onto the aluminum oxide surface followed by a few microns of hot (600°C) sputtered aluminum oxide. If any electrical shorting occurs through the sputtered aluminum oxide layer, the problem can be eliminated by heat treating in air at 700°C for 6 hours.
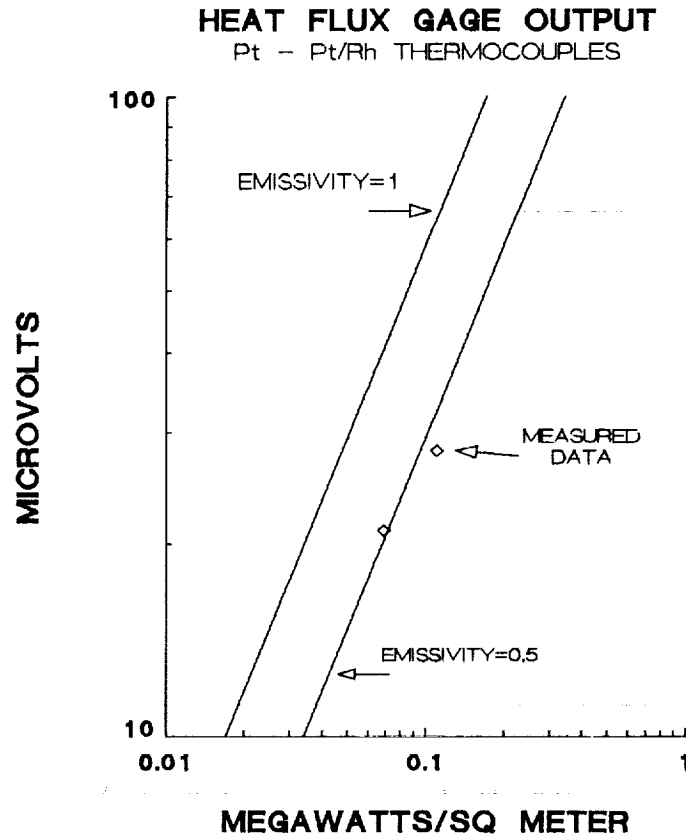

HEAT FLUX GAGE OUTPUT
Pt — Pt/Rh THERMOCOUPLES

EMISSIVITY=1

MEASURED DATA

EMISSIVITY=0.5

MICROVOLTS

MEGAWATTS/SQ METER

Figure 7. Calculated output of 20 element heat flux gage. Data points represent measured values.

DISCUSSION

The advantage of this heat flux sensor is that it provides a sensor that is minimally intrusive to gas flow, does not require machining of the mounting surface, and provides a relatively large output signal. The output can be ten to ten thousand or more times that of single temperature sensors. This multiplication of the output signal, by adding numerous thermocouple pairs in series, has the result of increasing the signal relative to the noise in the measuring system. This can be especially significant since thermocouple voltages are often in the microvolt range - a range where Johnson noise can easily mask the desired signal.

The heat flux sensor is fabricated from very thin films of metals and insulators. While thin films do not have zero mass, their mass is generally thousands of times less than the part they are

attached to. As a result the time constant, or speed of response, of the sensor will be extremely fast since the output only depends on heating up a film that is a few microns thick. It should be noted that the time constant for the temperature of the top or bottom surface of the heat flux sensor will be fairly slow - depending on the mass of the engine part. However the temperature difference across the sensor will respond very rapidly since this is only dependent on heating the thin film. At this time no calculations or measurements have been done to determine the time response.

The output of the heat flux sensor is a voltage generated by thermocouples. The voltage is linearly proportional to heat flux as was shown in a previous section. Thus heat flux is obtained by multiplying the output voltage by a constant.

The sensor has not been tested at cryogenic temperatures but it is expected to survive temperatures from below liquid O_2 to about 1700°C. The upper temperature is limited by the silicon dioxide insulator. The sensor shown in figure 2 makes use of platinum based thermocouples. As a result this sensor has a low temperature limit of 0°C. The low temperature limit can be extended by using other thermocouple materials. The use of chromel/alumel thermocouple alloys would allow a temperature range of -184°C to +1260°C.

Although the thin film heat flux sensor is difficult to fabricate it can provide the engine designer with much useful information about heat loading on engine components.

FUTURE WORK

The current work on the heat flux sensor will continue using silicon, aluminum oxide, and MAR-M-200[2] as substrates. The sensors will be tested in a heat flux calibration facility capable of generating heat fluxes in the range of 1 to 5 Mw/m^2. Fabrication techniques will continue to be refined.

All of the work on thin film heat flux sensors has concentrated on flat substrates. Most propulsion system components are not flat. It is a difficult problem to put a thin film pattern onto curved surfaces. Various methods for solving the curved surface problem are being investigated. One of the methods that will be tried is the use of flexible photomasks. A photomask fabricated on 127 micron (.005") Teflon[3] is shown in figure 8.
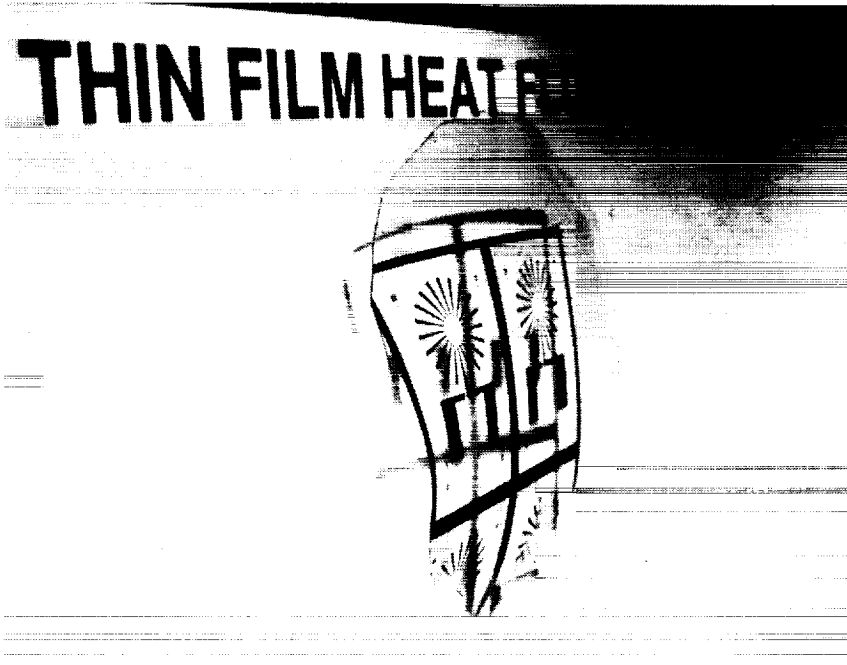
Figure 8. Flexible photomask fabricated on Teflon[3].

REFERENCES

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2.   Sims, Chester T.; Stoloff, Norman S.; Hagel, William C.: "Superalloys II", John Wiley & Sons, 1987, page 108.

3.   Teflon - Reg TM E. I. du Pont de Nemours & Co.