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13. ABSTRACT (Maximum 200 words)
This investigation concerns the development of microsensors sensors on flexible substrates such as Kapton (pyromellitic dianhydride and 4,4' diaminodiphenyl ether). Microbolometers have been used as the test bed for microsensor development. The results on this investigation will form a basis for the production of other micromachined sensors such as pressure/strain sensors, "hair-like" touch and flow sensors, and accelerometers on flexible substrates. Flexible substrates can serve as the basis of a sensitive skin for humans and robots where sensors are distributed over skin to provide the sense of touch and feel or monitor the physiology of the wearer. Two techniques have been investigated. One involves attaching a Kapton film to a 4-inch wafer carrier with an adhesive and the other involves using a spin-on polyimide (Kapton-like) to coat a silicon wafer that has been covered with a release layer. The use of 4-inch wafers permits the use of standard microfabrication equipment. Using these techniques, YBaCuO microbolometers have been fabricated. Low temperature fabrication techniques are employed to minimize the thermal cycling of the polyimide substrate. In our preliminary work, the microbolometers displayed a Temperature Coefficient of Resistance of -3.1 %, at room temperature. The microbolometers reached responsivity and detectivity as high as 6x10^3 V/W and 3x10^5 cm.Hz^{1/2}/W, respectively, at 1.85 μA of current bias. This detectivity is approximately 40% of the temperature fluctuation limit for these detectors.

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(4) Statement of the problem studied

This investigation concerns the development of suspended, micromachined sensors on flexible substrates such as Kapton (pyromellitic dianhydride and 4,4’ dianimodiphenyl ether), Ultem (Polyetherimid), PEEK (Polyetheretherkeytone), Supec (Polyphenylene Sulfide) and Mylar (polyethylene terephthalate). The results on this investigation will form a basis for the production of other micromachined sensors such as pressure/strain sensors, "hair-like" touch and flow sensors, and accelerometers on flexible substrates. Flexible substrates can serve as the basis of a sensitive skin for humans and robots where sensors are distributed over skin to provide the sense of touch and feel or monitor the physiology of the wearer. Thin-film Si or polymer-based transistors could provide read-out and signal processing functions. Micromachined microbolometers have demonstrated high sensitivity and room temperature operation. Micromachined thermal isolation structures allow microbolometers to integrate the heat received from the infrared radiation flux to produce a strong response. Semiconducting Yttrium Barium Copper Oxide (YBaCuO) microbolometers are fabricated by rf sputtering at ambient temperature. The only high temperature-processing step is the 250°C cure of the polyimide sacrificial layer. This makes YBaCuO compatible with many flexible substrate materials. Noise equivalent temperature differences less than 100 mK can be achieved for infrared detection even when operating at atmospheric pressure. Integrating low-density, staring microbolometer arrays over the surface of a flexible skin can allow a robot to remotely measure temperature and avoid hot objects that can cause damage. "Insect-like" vision can be achieved for the operator. The motion of hot objects such as people can be tracked. Gloves, which provide temperature-sensing abilities, can be produced combining YBaCuO microbolometers and YBaCuO thermometers in contact with the substrate. Although not a part of the SGER proposal here, in the long term, combining IR emitters with IR sensors on flexible substrates would allow fabrication of wearable infrared spectrometers to monitor environmental parameters such as toxic gases, and bacteriological agents and to monitor physiological parameters such as glucose and insulin levels.
Different flexible substrate candidates will be evaluated for compatibility with the micromachining process. The flexible substrates will be passivated with Si$_3$N$_4$ films. The Si$_3$N$_4$ passivation will provide the additional feature of planarizing some of the surface roughness of the substrate. The polymer substrates will be bonded to a Si wafer carrier to provide rigid support during microfabrication. The devices will be fabricated on one flexible substrate. A second "substrate" film will be bonded to the first to encapsulate the devices with a superstrate. The flexible substrate and superstrate films will be the same thickness so the device plane is a plane of minimal strain. Since the microbolometer will be suspended, a cavity (5-10 μm deep) will be etched into the top polymer superstrate that will protect the suspended structures. The application of a seeing skin would utilize low-density arrays with detectors spaced with a millimeter pitch to provide "insect-like" vision. The optical transmission characteristics of the superstrate material will be investigated and maximized.

The design rules and technology developed through this investigation will be applicable to a broad class of micromachined sensors that may be produced on flexible substrates. The connectivity of devices on flexible substrates will be investigated by evaluating the performance of conventional metal and poly-Si interconnects as well as polymer semiconductor interconnects. These interconnects will be evaluated on the basis of their mechanical properties, noise performance and current handling capability.

To the best knowledge of the PIs, this is the first ever effort to build micromachined structures for IR sensors on flexible substrates. This research is especially suitable for a STIR grant since it represents a high-risk research activity due to the challenges it faces in incorporating polymers in a standard CMOS fabrication facility. The pay-off is very high due to the immense number of applications that "sensitive smart skin" has. A major obstacle in the development of vanadium oxide bolometers or lead tinate and similar oxide pyroelectric detectors on flexible substrates is the incompatibility between the thermal budget required by the detector material and the low maximum temperature of the flexible substrate. This proposal will solve the incompatibility between the two thermal budgets by the use of the PI's patented material: semiconducting Yttrium Barium Copper Oxide (YBaCuO). The deposition temperature for this IR sensitive material does not exceed 150 °C. High quality nanocrystalline, semiconducting YBaCuO films can be deposited using rf sputtering at ambient temperature. Since there is no requirement for crystallization, high-temperature annealing steps are not necessary. The activities proposed here will provide the foundation for a new technology of smart skin.

(5) Summary of the most important results

I. INTRODUCTION

The performance of silicon technology is not sufficient to satisfy the demand for new novel technologies in which mechanical flexibility is often essential, such as foldable electronics. To meet these technology requirements, a flexible substrate must be used. Unlike silicon, flexible substrates are usually more robust, lighter weight, and offer mechanical flexibility to absorb the stress. On the other hand, the nature of the flexible substrates introduces processing and material challenges such as maintaining flatness of the film during fabrication process and fixture tooling, employing electronic materials that can be flexed without degradation or failure. In addition, flexible substrates can experience considerable plastic deformation from thermal cycling. To maintain flatness, the flexible substrate may be attached onto an ordinary silicon wafer temporarily and be removed after fabrication. This allows conventional silicon processing equipment to be used for device fabrication. Two techniques have been investigated. The first involves attaching a Kapton film onto a Si wafer carrier with an adhesive, while second uses a spin-coated liquid Kapton-like polyimide. The later technique has shown the most promise in avoiding plastic deformation of the substrate during the microfabrication of the devices, though substrate removal and the effects of subsequent flexing and deformation require further investigation.

In this investigation, Kapton, a commercial name of a specific polyimide film produced by DuPont is used as a flexible substrate. Kapton belongs to the group of polymers demonstrating excellent chemical, mechanical and electrical properties as well as high thermal stability. It has a temperature operation range of -269 to 400 °C. These advantages of the flexible substrate Kapton together with high sensitivity of Yttrium-Barium-Copper-Oxide (YBCO) bolometers operating at room temperature form the basis of this design. YBCO is also attractive for its room temperature deposition by rf magnetron sputtering. As-deposited YBCO films display low inherent 1/f noise, good infrared absorption properties, and a relatively large temperature coefficient of resistance (TCR ~ -3.5%/K).

The flexibility of polyimide substrates can lead to new applications and extend the reach of bolometers into new areas, such as smart skins for robotics, wearable body monitoring systems, smart gloves, smart tags, and curved matrix arrays for IR imaging. Bolometers are also typical microsensors that are fabricated by using microfabrication
and micromachining techniques. In this respect, the fabrication techniques and results of this investigation have a broad impact to microsensors and other devices fabricated on flexible, polymer substrates.

A bolometer is a device for detecting and measuring optical radiation. The theory of the electrical and thermal characteristics of bolometers has been discussed in detail elsewhere[1]. A bolometer is a device whose electrical resistance changes with temperature and therefore acts as a thermometer. The power absorbed from incident optical radiation heats the thermometer, providing a means of measuring the radiant energy flux. The TCR of the bolometer is an important figure of merit in determining the relative magnitude of the response. TCR is defined as [2,3,4,5,6]:

$$TCR = \alpha = \frac{1}{R} \frac{dR}{dT}$$  \hspace{1cm} (1)

where $R$ is the resistance and $T$ is the temperature. In this research, a semiconducting YBCO was used which has a relatively higher TCR of $-3.5\%$, compared to its counter parts, around room temperature, such as VOx [4] or metal film bolometers [7].

Heat lost from the bolometer to its surroundings through radiation, convection and conduction limits the responsivity. Among these, the conduction of heat, through substrate and electrode arms dominates. Hence, micromachined devices isolated from the substrate have lower thermal conductivity, $G$, and increase the responsivity in vacuum. The conduction to the substrate is a function of modulation frequency of incident optical radiation [8]. The change in the output voltage from a bolometer due to temperature change of $\Delta T$ is given as $\Delta V = IRe\Delta T$ where $I$ is the bias current. The responsivity is defined as the output voltage signal per watt of incident radiation power falling on the pixel, and is given as [9]:

$$R_s = \frac{R_{T2}}{A_0K_\omega L_2C_2\omega}$$ \hspace{1cm} (2)

in the case of strong thermal coupling to the substrate. Here, $K_s$ is the thermal conductivity, $C_2$ is the heat capacity per unit volume of the substrate, $\omega$ is the optical modulation frequency and $\eta$ is the optical absorption coefficient of the thermometer and $A$ the detector area.

The actual measure of a detector performance depends on how good it can extract a signal from the surrounding noise. The specific detectivity can be expressed as [1,2]:

$$D^* = \frac{R_s\sqrt{A\Delta f}}{V_n}$$ \hspace{1cm} (3)

where $V_n$ is the total noise voltage observed in the electrical bandwidth $\Delta f$. The specific detectivity determines the ability of a detector to resolve changes in optical power and noise is the limiting factor for the bolometers specific detectivity.

The noise figure of the IR measurement setup is composed of five components. These are background noise originating from radiating heat exchange between the pixel and its surroundings; phonon noise due to interaction with heat sink; Johnson noise of the bolometer, that of the preamplifier and the $1/f$ noise. Among these components, $1/f$ noise dominates at low frequencies, whereas the Johnson noise governs at high frequencies.

**II. DEVICE STRUCTURE AND FABRICATION**

Arrays of 1x10 microbolometers with lateral dimension of 60x60 $\mu$m$^2$ were fabricated on Kapton polyimide films using semiconducting YBCO thin films. Two techniques were employed in the microfabrication were employed. The first involved attaching a Kapton film to a Si wafer carrier with and adhesive. The second used a spin-on, Kapton-like, polyimide substrate that was later released from the Si wafer by using a release layer. The flexible nature of the Kapton substrate provides special challenges especially for dimensional control. The later technique provided good dimensional stability during the fabrication process. In developing the processing techniques the following concerns of flexible substrates were addressed. Each processing step was examined and every effort made to sustain repeatable fabrication control in order to minimize any substrate variation. The substrate needed to be adhered to a Si wafer carrier in a means that maintained flatness of the Kapton during film deposition and lithography. The temperature of processing steps was considered to minimize thermal plastic deformation of the Kapton, which would result in alignment problems during subsequent photolithography steps.

In the first technique investigated, the microbolometer fabrication started with adhesion of a 125-um-thick Kapton film onto a 4-inch Si wafer carrier using a spray-type adhesive. Several different adhesives were tried in this investigation. The adhesive with the best properties was found to be a commercially available adhesive 3M Super 77. The top surface of the Kapton substrate was passivated with an 8500 A Si$_3$N$_4$ film, using RF magnetron
sputtering in 10 mTorr Ar and ambient temperature. This layer also served as a barrier against humidity absorption by Kapton, which is 3% [10]. In order to enhance the absorption of incoming IR, a 4000 Å thick Al mirror layer was introduced next. This mirror layer forms an optical microcavity with the microbolometer thermometer. The next step was to deposit a thermal and electrical isolation layer upon which the IR sensitive detectors could be constructed. For this purpose, a liquid polyimide (PI2610) was selected. In addition, the polyimide can also be used as a sacrificial to achieve higher thermal isolation later in the investigation. Commercially available PI2610 was spun onto the structure at 3200 rpm for 30 seconds followed by a soft bake at 135 °C for 30 minutes. The polyimide was cured at 270 °C for 75 minutes in N₂ ambient, yielding a thickness of about 2 µm. This is believed to be the highest temperature the wafer was subject to during the microbolometer fabrication. It should be noted that the plasmas used during sputtering, dry etching, and ashing procedures also heat the wafer. In this case, the poor thermal conductivity of polyimides such as Kapton establishes a thermal gradient across the thickness of the wafer with relatively high temperature achieved on the top surface. Careful consideration was given to ensure low temperature processing such thermal deformation of the Kapton substrate is avoided.

The microbolometer electrode arms utilized a 1000 Å thick Au layer sputtered film patterned by conventional photolithography and wet etching with a KI/I₂ solution. The last deposition step was to sputter a 4000 Å YBCO thin film at room temperature [11]. The YBCO thermometers were patterned by wet etching with a 1:100 aluminum etch solution at room temperature. At this point, the microbolometer fabrication process was complete and the Kapton substrate was removed from the carrier Si wafer. Individual dies were attached on to a ceramic flat package and test devices were ultrasonically bonded using Au wires. Fig. 1 shows a Kapton substrate with completed microbolometers being flexed. The monitor in the background displays a 1x10 array. The Au electrode arms and bond pads are the most prominent features recognizable on the monitor. The Kapton substrates could be bent without damaging the microbolometers, though the limits of flexibility have not been investigated quantifiably. Fig. 2 shows a SEM picture of a microbolometer constructed on 125-µm-thick Kapton substrate.

The fabrication process for the second type microbolometers on the liquid Kapton substrate was similar to that of the first type. Fabrication process started with sputtering of 800 Å MgO as a release layer on Si wafer. Next, liquid Kapton was spun onto the wafer in three steps and hard baked yielding a total thickness of approximately 24 µm. Having sputtered the same thickness of Si₃N₄, metallization was completed with a 2000 Å Ti followed by a 1000 Å Au. After lithographically defining the metal electrode arms, YBCO with a thickness of 2000 Å was sputtered on the structure and patterned to obtain IR sensitive regions. The Kapton substrate was released from the Si wafer by etching the MgO release layer with acetic acid.

In contrasting the devices fabricated by these two techniques. In the first technique, the adhesion of the Kapton film to the Si wafer was not perfect. Small bubbles became apparent as the wafer was processed. In addition, the adhesion was not strong enough to prevent the Kapton film from plastic deformation during the device fabrication. The plastic deformation is estimated to be 0.01% or less, but this is significantly large to result in misalignments across the 4-inch wafer. The adhesive was relatively thick, permitting rapid removal of the Kapton substrate from the wafer carrier. In the second case, the spin-on substrate was strongly attached to the Si wafer carrier. There was no distortion observed in the polyimide substrate during the microbolometer fabrication allowing for accurate lithographic patterning of the device structure. The release of the polyimide substrate from the Si carrier was relatively slow due to the thin MgO release layer employed. The speed of release is expected to increase with a thicker release layer.

Figure 1. A photograph of a completed die with microbolometers on 125-µm-thick Kapton substrate.
III. RESULTS AND DISCUSSION

After packaging the individual die, bolometers were tested. The two 60 μm x 60 μm microbolometers characterized had a dc resistance of 1.14 and 1.60 MΩ at room temperature and they displayed linear I-V characteristics up to 2 μA. The TCR value was determined through Eq. (1) from an R-T measurement between 220 and 320 K, in a cryostat at a pressure of 50 mTorr. It is found to be approximately −3.1 % at room temperature. This is a very typical value for a semiconducting YBCO [2,11]. The measured R-T and calculated TCR graph can be seen in Fig. 3.

![Graph showing temperature dependence of resistance and corresponding TCR](image)

Figure 3. Temperature dependence of resistance and corresponding TCR for a microbolometer on Kapton, over the temperature range of 220 to 320 K. TCR was obtained through Eq. 1.

Joule heating method was used to find out the thermal conductance, G, which was evaluated from the slope of the resistance versus power characteristic and found to be 4.18x10⁻² W/K [12]. This is a relatively low G value for a bolometer on the substrate. This can be attributed to the low thermal conductivity of the polyimide film and
Kapton substrate. The theoretical calculation of the same value, ignoring the thermal boundary resistances, yielded a very similar value of 2.94×10^{-3} W/K. The thermal conductance values through the electrode arms of 1000 Å thick Au and through the substrate are very similar in magnitude. At this point, the stability of electrode arms can also be discussed. Adhesion of metal to polyimide is a key performance requirement for polyimide flexible substrates. Although a good initial adhesion is essential, adhesion retention after thermal and humidity stress are as important. Any of these stressing conditions have the potential of reducing adhesion, causing process yield losses. Gold with a thermal expansion coefficient of 14.2×10^{-6} K^{-1} was subjected to stress due to the different thermal expansion coefficient of polyimide, 17-32×10^{-6} K^{-1}. Yet, gold adhered well to the polyimide.

The responsivity of the bolometers was characterized using a chopped radiation from a 1450 K IR source. The results were calibrated with an Oriel 70124 pyroelectric detector. The responsivity measurements were done in air and vacuum at 60-mTorr pressure at room temperature. Results showed no dependence on ambient pressure as expected because the detectors were not micromachined and in full contact with the substrate. The responsivity had a $1/\sqrt{\omega}$ dependence as expected from Eq.(2). Fig. 4 shows the responsivity of a detector on Kapton substrate. The detectivity characteristics corresponding to the relevant responsivity at different current biases is given in Fig.3(b). While the background limited specific detectivity of this detector had a value of 8×10^7 cm Hz^{1/2}/W, the measured detectivity reached up to 3×10^8 cm Hz^{1/2}/W as limited by the $I/f$ and Johnson noise at low and high chopping frequencies, respectively.

Responsivity and detectivity measurements for the second type microbolometers were completed without removing the Kapton substrate from Si wafer. Results yielded a responsivity and detectivity as high as 350 V/W and 7×10^8 cm Hz^{1/2}/W at 1 μA, respectively (See Fig.5).

![Graph](image)

**Figure 4.** Responsivity and detectivity of the microbolometer on Kapton as a function of chopper frequency at four different current biases, in vacuum. The straight line in (a) shows $1/\sqrt{\omega}$ dependence of responsivity.
The absorption coefficient, $\eta$, was calculated from the best fit of the measured responsivity to the theory. For this calculation, heat capacity per unit volume, $C_v$, 1.521 J/Kcm$^3$; and thermal conductivity, $K_v$, 1.045x10$^3$ W/cmK of the polyimide [13] and measured TCR were used. The best fit for two different devices on Kapton gave values of 22% and 15% for $\eta$, which is very typical and promising without an absorber layer on top.

The possible substrate effect was investigated with a surface scan using a pinhole optical source. Detector was attached on to a 3D controlled plane. Then, it was illuminated through a pinhole with a diameter of 444 $\mu$m at a distance of 2.3 mm. The device plane was scanned by 125 $\mu$m steps and output voltage signal was recorded while IR light was chopped at 9 Hz. The signal was observed from an area of about 760 $\mu$m in diameter whereas the calculated illumination area was 560 $\mu$m in diameter. The difference was attributed to the scattering and diffraction effects from the edges of the aperture. The Fig 6 implies that there was either no contribution from the substrate or it was negligible.

Figure 6. Surface scan of a single pixel on Kapton substrate with 125-$\mu$m-step size. No signal was observed unless the pixel was illuminated.
IV. CONCLUSION

In this work, YBCO based microbolometers were fabricated on both Kapton® and liquid Kapton. For the first type detectors, the TCR value at room temperature, -3.07%, was in an agreement with previous values for semiconducting YBCO. The characterization of microbolometers yielded a promising responsivity of $6.12 \times 10^3$ V/W at 1.84 $\mu$A current bias, partially due to the achievement of a relatively high thermal isolation (G=$4.18 \times 10^5$ W/K), using a polyimide layer with a low thermal conductance under the pixel. As a result, the detectivity reached up to $3 \times 10^7$ cm Hz$^{1/2}$/W. This is a very promising result considering that this is the first attempt to build room temperature microbolometers on a flexible substrate.

The microbolometer response versus IR radiation modulation frequency was found to imply a substrate coupling with $1/\sqrt{\omega}$ dependence. The absorption coefficient was 15% and 22% for 2 different devices. Although micromachining was not attempted for this first generation microbolometers, a surface scan test proved that the response was a pure bolometric response and not from the substrate.

For the second type of detectors, responsivity and detectivity gave values of 350 V/W and $7 \times 10^6$ cm Hz$^{1/2}$/W at 1 $\mu$A, respectively. This research was a successful first attempt to fabricate micromachined bolometers on a plastic substrate. The difference in the responsivity and detectivity of the first and second types of microbolometers is attributed to the higher thermal conductance of the thinner polyimide substrate in the second case.

At the time of this report, micromachined microbolometers have been fabricated, but they have not been characterized yet.

(6) Listing of all publications and technical reports supported under this grant or contract. Provide the list with the following breakout, and in standard format showing authors, title, journal, issue, and date.

(a) Papers published in peer-reviewed journals

(b) Papers published in non-peer-reviewed journals or in conference proceedings

(c) Papers presented at meetings, but not published in conference proceedings


(d) Manuscripts submitted, but not published


(e) Technical reports submitted to ARO

(7) List of all participating scientific personnel showing any advanced degrees earned by them while employed on the project

Professor Donald P. Butler

Professor Zeynep Celik-Butler

Mr. Alp Yaradanakul (Ph.D. to be received shortly)

Mr. Ali Yildez (Ph.D. to be received shortly)

(8) Report of Inventions (by title only):

1. MgO Release Layer Layer for Substrate Transfer and Carriers for Flexible Substrates (Invention Disclosure to be Filed)
(9) Bibliography


(10) DuPont Bulletin H-12 Kapton Polyimide Film.


(10) Appendixes: None