High T_c superconducting bolometer on chemically etched 7μm thick sapphire

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Abstract

A transition-edge IR detector, using a $YBa_2Cu_3O_{7-x}$ (YBCO) thin film deposited on a chemically etched, 7 μm thick sapphire substrate has been built. To our knowledge it is the first such high Tc superconducting (HTS) bolometer on chemically thinned sapphire. The peak optical detectivity obtained is $1.2x10^{10}$ $cmHz^{1/2}/W$ near 4Hz. Result shows that it is possible to obtain high detectivity with thin films on etched sapphire with no processing after the deposition of the YBCO film. We discuss the etching process and its potential for micro-machining sapphire and fabricating 2-dimensional detector arrays with suspended sapphire membranes. A 30 μm thick layer of gold black provided IR absorption. Comparison is made with the current state of the art on silicon substrates.

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Introduction:

A lot has been written on the fact that transition-edge, high temperature superconducting (HTS) bolometers [1,2,3] perform better than photon detectors at temperatures above 77K and at wavelengths above 20 μm. For space borne instruments which carry detectors cooled to LN2 temperatures and are looking at the far IR part of the spectrum ($\geq$ 20μm), high Tc bolometers would show better performances than thermopiles and other room temperature detectors currently used [4].

Its strength and its high Debye temperature make sapphire a material of choice, for bolometer fabrication. The deterioration of HTS thin films on silicon substrates reported in the literature [5] combined with the large thermal expansion coefficients mismatch between HTS films and silicon raise questions about the long term stability of silicon based HTS bolometers. Until these issues are resolved conclusively, we believe that alternative methods (i.e. sapphire based detector development) should be pursued.

Additionally, if one compares the heat capacity values of sapphire and silicon at 90 K, sapphire's heat capacity, per unit volume, is about 33% lower than silicon's [6]. The fabrication technique we have used is simple, requires only a few steps, requires no passivation layers against further processing steps and requires no impedance matching of the thermometer with an antenna coupled to it [4]. No etching or patterning is needed either, after the deposition of the HTS thin film, which can introduce damages in the film. And yet an optical detectivity above 10^{10} $cmHz^{1/2}/W$ near 4 Hz has been achieved..

The bolometer can be modeled as a substrate with a heat capacity C heat sunk via a thermal conductance G. In the case of a current biased bolometer the responsivity, S, can be expressed as follows:

$$S = (IdR/dT)/G\ (1+\omega^2\tau^2)^{1/2} \qquad (1)$$

Where I is the bias current, ω is the angular frequency of the modulated signal, dR/dT is the slope of the detector's resistance versus temperature, at the transition. τ = C/G is the response time.

The electrothermal feedback or the electrical dissipation in the thermometer due to the bias applied reduces the thermal conductance G to an effective G_{eff} in a the following manner:

$$G_{eff} = G - I^2 dR/dT \qquad (2)$$

After our previous reported work [4] we started an effort to reduce first the heat capacity of the sapphire substrate by reducing its thickness. And, at a second stage, to minimize the thermal conductance and obtain the desired sensitivity and response time. The bolometer built then, achieved a peak detectivity $D^* = 6.9 \times 10^9$ cm/W.$Hz^{1/2}$ at 4 Hz. The bolometer's thermometer element was a YBCO thin film grown on a mechanically polished, 25μm thick, sapphire substrate by Pulsed Laser Deposition (PLD). The G_{eff} obtained was about 10^{-4} W/K. And the time constant was 65 ms at low bias and a heat capacity of 6.5×10^{-6} J/K.

Design and fabrication:

For the bolometer discussed in this paper, the substrate dimensions are $1mm^2$ X 7 μm. Two Kevlar fibers provide the suspension as well as thermal isolation. A thin buffer layer of CeO_2 is deposited on one side of the substrate over which the YBCO thin film thermometer is grown. Electrical contact is provided by four thin gold wires. The latter also provide the thermal conductance to the heat sink. A low filling-factor gold black layer provides IR absorption. It is deposited on the side of the substrate not covered by the YBCO thin film.

Chemical etching:

To reduce the heat capacity of the sapphire two methods were tried. One involves ion milling of single crystal sapphire and another involves chemical etching. Three 12 μm thick wafers were obtained by ion milling. Both CeO_2 and YBCO layers were grown over these thin wafers by PLD. A modified blackbody type heater was used to avoid damaging the wafers [7].

However, it was discovered later that, unless the samples are well thermally sunk, ion milling produces an amorphous, opaque and vitrified layer. The latter behaves as amorphous sapphire with degraded thermal properties. On the other hand, a chemical etching process, developed by Reisman et al. [8], was used for thinning the substrate and it produced no opaque layer. It relaxed the tensile stress in the wafers which became flatter (as opposed to slightly bent) and very transparent.

The etching procedure was customized to our requirements by using suspended Teflon holders to keep the wafers perpendicular in the etching solution. Hence, single crystal, R-plane sapphire wafers (1102 orientation), with thicknesses as low as 7 μm were obtained. We know now that we could have continued the etching to obtain 1 μm thick (or less) sapphire wafers which are probably still strong enough to withstand the whole detector fabrication process.

Five 25 μm thick, 1/2 inch diameter, R-plane sapphire wafers were etched in a 1:1 mix of concentrated H_3PO_4 and H_2SO_4 at 300°C. A range of thicknesses from 14 to 7 μm were obtained. Microscopic

examination of one of the samples showed that it was an isotropic etch. The etch rate was on average 6 to 10 μm/hour. Thicknesses were measured by comparing wafers' weights before and after etching and also by using a calibrated microscope-camera-IPLab image grabbing system. One of the etched wafers was analyzed under a high magnification microscope. It showed no particular and discernible change in the surface appearance and morphology due to the chemical etching. X-ray diffraction analyses of surface structures was also performed on a control sample before and after etching. Results showed no difference between the two. Fig 1. summarizes the findings.

Thin film deposition:

Growth of epitaxial YBCO/CeO_2 thin film structures on the thinned R-plane sapphire substrates was performed at Neocera, Inc [9]. The YBCO films and CeO_2 buffer layers were deposited *"in situ "* by PLD.

Details of the process have been reported earlier [7]. Briefly, a 20 nm thick CeO_2 buffer layer was deposited at 900 °C in an oxygen partial pressure of 300 mTorr. Then the temperature and the oxygen

pressure were lowered to 780 °C and 250 mTorr, respectively, and a 200 nm thick YBCO layer was grown. The sample was then cooled down to room temperature in an oxygen pressure of 1 atmosphere.

Thin film characterization:

The deposited YBCO films were characterized using *ac* susceptibility and x-ray diffraction. The films showed superconductive transition temperatures around 87 K with transition widths of $\leq$ 0.5 K. Fig. 2 shows the *ac* susceptibility plot from one such film. Using a Siemens D5000 four-circle x-ray diffractometer, θ-2θ, w and Φ scans were performed in order to determine the structural quality of one of the films. Results showed that the YBCO films were c-axis oriented with no secondary phases. In addition, the w and Φ scans indicated a high degree of c-axis alignment and in-plane epitaxial structure respectively. Overall, the x-ray results showed that these films have a high structural quality with no visible signs of high angle grain boundaries. The above YBCO film properties are essential in order to achieve HTS bolometers with low noise levels as well as stable and reproducible performance.

Detector assembly:

150 - 200 nm thick Ag contacts were thermally evaporated on three wafers using an Edwards System E306A at 5.5 X 10^{-5} torr. Then 15 nm thick gold was deposited on top of the silver contacts. This work was done at the University of Maryland [10]. Afterwards, the samples were annealed for 3 hours in O_2 at 500 °C. The wafer which showed the highest transition temperature (near 87 °K) and had the smallest thickness (7μm) was then diced to obtain 1 mm^2 pieces with four Ag/Au contacts. Several of the pieces were then gold bonded with 17.5 μm diameter and 1.5 mm long gold wires. Each was tested for highest Tc and one was chosen to build the HTS bolometer.

Approximately 30 μm of low filling factor [11] gold black was then deposited on the side of the sapphire substrate which has no YBCO, using NASA/Goddard's gold blackening facility.

Results:

Measurements were made by putting the bolometer in an evacuated liquid nitrogen cooled dewar with a KRS-5 window. The infrared source was a chopped blackbody at 500°C. The bolometer was coupled

to a low-noise preamplifier through a 1:100 transformer achieving a baseline noise of 0.5 $nVHz^{1/2}$. The four wire, low bias, resistance versus temperature was obtained and the peak dR/dT was found to be 2.5 Ω/K near the midpoint of the transition (3.5 Ω). The measured time constant(at low bias) was near 100ms.

The destabilization current was found to be I_{dest}= 8.75 mA. Plugging I_{dest} in equation (2) making G_{eff} = 0 and solving for G gives an implied G of about 1.9 X 10^{-4} W/K. This would suggest a heat capacity of 19x10^{-6} J/K.

Peak signal-to-noise (S/N) was obtained for a constant bias current of 6.35 mA (3/4 X I_{dest}). The measured D* at the midpoint of the transition was 1.2 X 10^{10} $cmHz^{1/2}/W$ up to 3.8 Hz with a corresponding optical NEP of 8 X 10^{-12} $W/Hz^{1/2}$. D* is shown in Fig. 3 after smoothing is performed to minimize statistical fluctuations due to microphonics.

Results are summarized in table No. 1

Discussion:

The upper limit for the thermal conductance can be estimated by assuming that the temperature noise is the main source of noise and neglecting the other sources. In this case [1]

$$NEP = (4k_B T^2 G)^{1/2} \qquad (3)$$

Since the measured NEP is 8×10^{-12}, then solving for G in equation (3) we have $G \leq 1.4 \times 10^{-4}$ W/K at $T = T_c = 90$K.

The G inferred from equation (2) is 1.9×10^{-4} W/K. The two values are close and indicate that we are very close to the theoretical lower limit of the NEP for the thermal sinking technique we use.

An inventory of the heat capacities of the different materials involved shows that the total heat capacity of the detector element (mainly the sapphire substrate, four gold wires and four gold balls at the gold/Ag contacts) is near 8.5×10^{-6} J/K at 90 K. The gold contributing 70% of the total heat capacity. The expected time constant is then 44 ms at low bias.

The solder used to connect the gold wires to the connector pins could potentially be a contributor of the remaining excess heat capacity. It is difficult to thermally model such a system and explain the higher heat capacity and time constant.

In the future, our plan for reducing the time constant is to avoid the use of gold wires altogether. Chemical etching, with the use of appropriate masks, could be used to create suspended 1μm thick (or lower) sapphire membranes. Two sapphire beams would provide the suspension. The beams would have the same thickness as the membrane. Electrical contact could be made via four ion-induced, CVD deposited, $\leq$ 1μm wide metal lines such as platinum.
We estimate that thermal isolation of the order of 1.2×10^{-5} W/K (for two suspension beams) can be achieved as well as time constants of the order 15 ms or below. The gold wires as well as the contact pads would be eliminated. One dimensional as well as two-dimensional arrays of HTS bolometers on sapphire membranes can also be envisaged in the very near future.

As in our previous bolometer, D* decreases at lower frequencies due to 1/f noise rising faster than the signal. Similar decrease is observed toward higher frequencies due to the signal falling faster than the noise.

Conclusion

In conclusion, we have developed a highly sensitive IR bolometer on a sapphire substrate thinned by chemical etching. Its fabrication involved no chemical processing after the deposition of the YBCO thin film. Its optical D* is 1.2×10^{10} $cmHz^{1/2}/w$ near 4 Hz. The best in the field has an optical D* of $1.8x\text{-}10^{10}\, cmHz^{1/2}/W$ from 0.2 to 2Hz and uses GdBaCuO thin film on a silicon substrate [12]. Our bolometer's D* is only a factor of 1.5 away and its peak detectivity is at a higher frequency.

In the near future, chemically etched one and/or two-dimensional arrays of sapphire membranes (less than 2 μm thick), suspended with sapphire beams should improve the performance of our bolometers even more.

Replacing the Yttrium in the YBCO compound with Gadolinium (Gd) may also lower the noise floor and increase the performance of our sapphire based bolometer since the literature [12,13] suggests lower noise with Gd compounds.

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Table 1.

	Previous bolometer[4]	Current bolometer
Al_2O_3 thickness(μm)	25	7
Heat capacity$_{detector}$(J/K)	6.5×10^{-6}	19×10^{-6}
Time constant$_{low\ bias}$(ms)	65	100
D^* cmHz$^{1/2}$/W	6×10^{9} @ 4 Hz	1.2×10^{10} @3Hz
NEP W/Hz$^{1/2}$	1.6×10^{-11}	8×10^{-12}

Captions:

Fig. 1 (a): X-ray θ~2θ scan of control sapphire wafer before etching.

Fig.1 (b): X-ray θ~2θ scan of same sample after thinning by chemical etching. The two major peaks match the reflections belonging to the (1102) family of planes of single crystal alumina.

Fig. 2: *ac* susceptibility data for YBCO thin film on deposited on chemically thinned R-plane single crystal sapphire.

Fig. 3. Detectivity obtained (bias current is 6.35 mA) and at midpoint of the transition.

Table 1. Summary of results - Performance of previous bolometer on mechanically polished sapphire substrate vs current bolometer on chemically etched sapphire substrate.

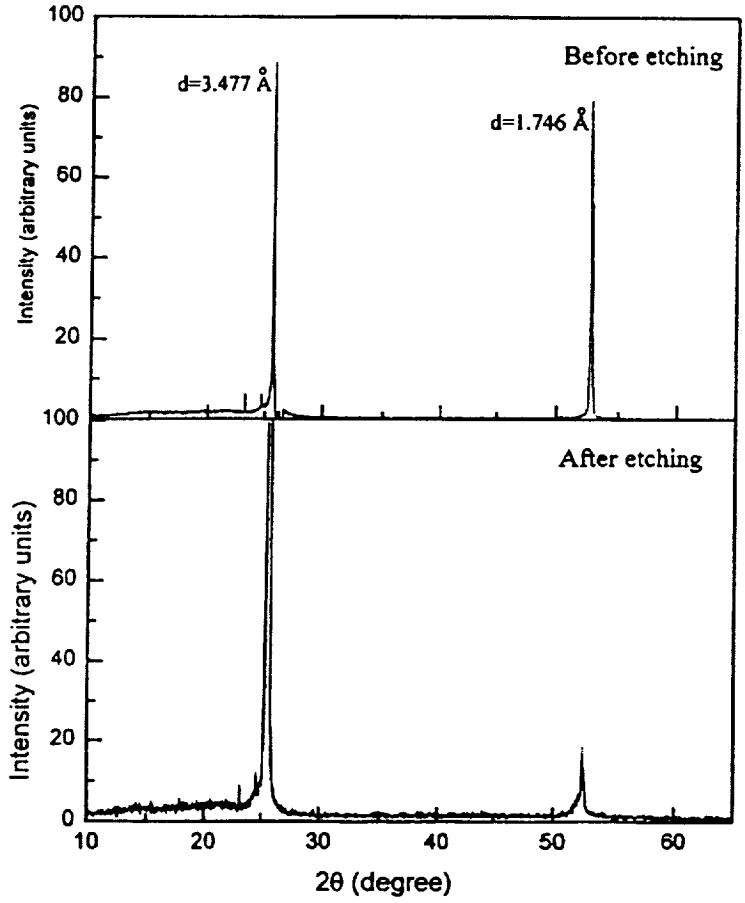
Before etching
d=3.477 Å
d=1.746 Å
After etching
Intensity (arbitrary units)
Intensity (arbitrary units)
2θ (degree)
100
80
60
40
20
0
10
20
30
40
50
60

fig.2
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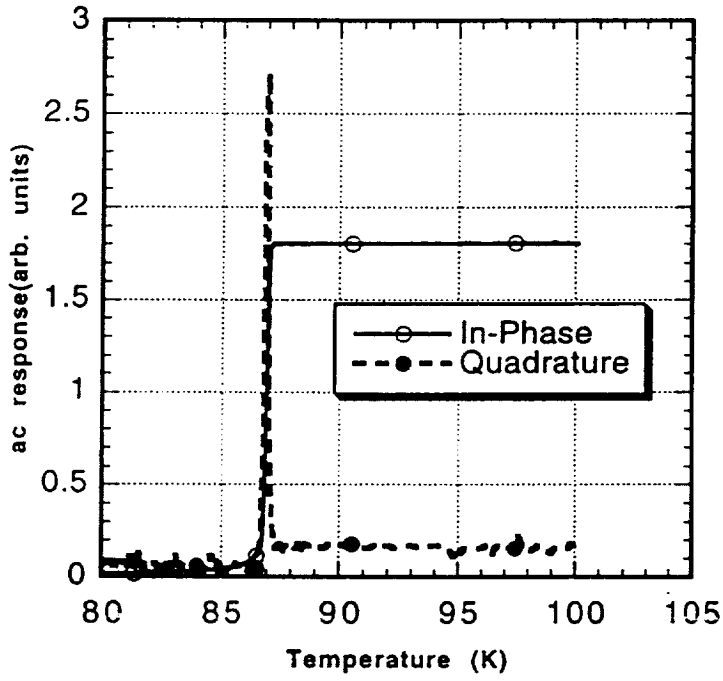

Fig. 3
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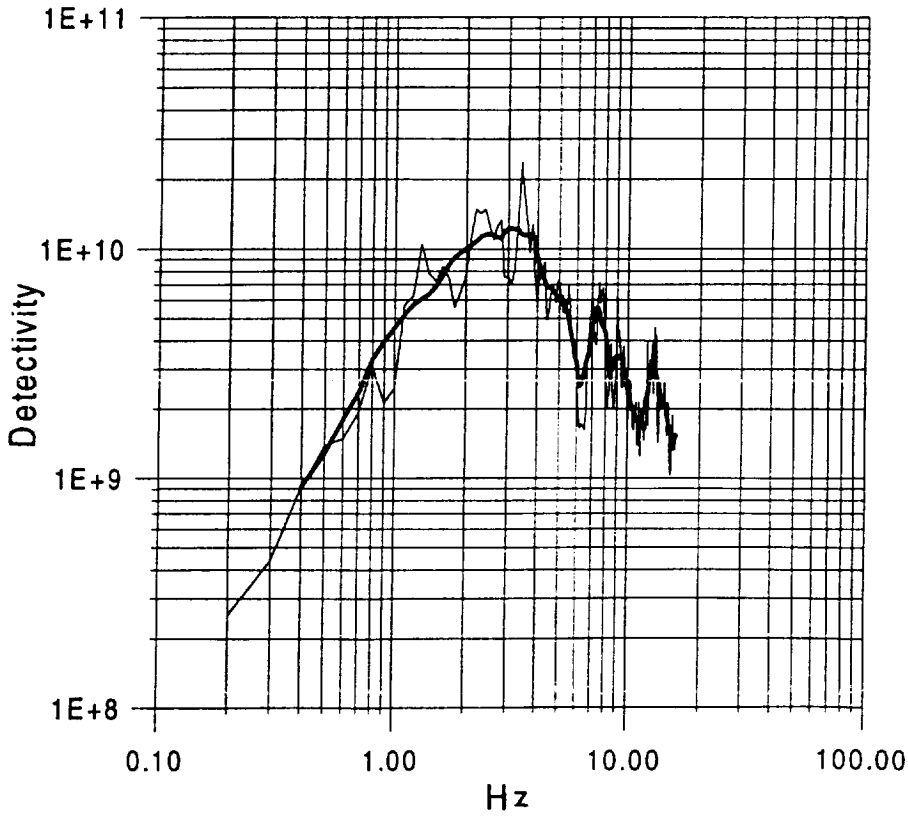

Fig. 3
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