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The IBATCE system will be used to initiate an extensive research on oxide materials, such as fabrication of HTS bolometer infrared detectors, through collaboration with DoD laboratories and other institutions. The driving force for this research is the DoD's need for high-sensitivity infrared detectors in long-/very-long wavelength ranges. The focus of the proposed research is to understand the growth mechanism of the HTS multilayer-structured bolometer infrared detectors in IBATCE process and to obtain high device performance by optimizing the thin-film quality and device designs. The long-term goal of this research is to build a competitive project in Kansas in the emerging field of oxide materials and to bring in significant amount of federal research funds. The requested DEPSCoR fund is crucial to the success of the proposed research and such a success certainly benefits the State of Kansas economy and meets the goal of DEPSCoR program.

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*Final Report*

**Fabrication of high- $T_c$  superconducting infrared detectors using ion-beam-assisted thermal co-evaporation**

***Abstract***

This proposal requests equipment funds to construct a novel ion-beam-assisted thermal co-evaporation (IBATCE) system. The IBATCE aims at resolving critical issues identified in fabrication of multilayer-structured devices comprising a large variety of oxide materials including high- $T_c$  superconductors (HTSs), ferroelectric, pyroelectric, colossal magnetic materials, etc. Since most of these materials are highly anisotropic, epitaxial growth of thin films is desirable in order to optimize physical properties associated with certain crystalline orientation and to minimize the noise caused by large-angle grain boundaries. Growth of **crystalline epitaxial** oxide materials on **polycrystalline/amorphous** metal electrodes or semiconductor-based readout integrated circuits at low growth temperatures with large-area uniformity and high growth rate, therefore, becomes a stringent requirement in the device application of oxides. Such a requirement cannot be fulfilled in most thin film deposition processes currently available. Despite the promising perspective the oxide materials offer in microelectronic applications, little progress can be made without solving these problems. By combining the state-of-arts ion-beam-assisted deposition (IBAD) with the industrially proven thermal co-evaporation (TCE) techniques, the IBATCE provides an unique solution to the problem, enabling achievement of high device performance and high device throughputs.

The IBATCE system will be used to initiate an extensive research on oxide materials, such as fabrication of HTS bolometer infrared detectors, through collaboration with DoD laboratories and other institutions. The driving force for this research is the DoD's need for high-sensitivity infrared detectors in long-/very-long wavelength ranges. The focus of the proposed research is to understand the growth mechanism of the HTS multilayer-structured bolometer infrared detectors in IBATCE process and to obtain high device performance by optimizing the thin-film quality and device designs. The long-term goal of this research is to build a competitive project in Kansas in the emerging field of oxide materials and to bring in significant amount of federal research funds. The requested DEPSCoR fund is crucial to the success of the proposed research and such a success certainly benefits the State of Kansas economy and meets the goal of DEPSCoR program.

## *Description of Proposed Research*

### **1. Introduction**

This proposal requests equipment supports to the experimental research program at the University of Kansas (PI: Judy Wu). A total amount of \$170k is necessary to construct a novel ion-beam-assisted thermal co-evaporation (IBATCE) system, aims at solving the critical problems in fabrication of high- $T_c$  superconducting bolometers compatible with commercialization of high-sensitivity infrared detectors. Since an institutional matching fund of \$16k and a state matching fund of \$42k will be provided for the proposed research, a total of \$112k fund is requested from the DEPSCoR program.

#### **1.1 Backgrounds**

Infrared (IR) detection has been extensively investigated ever since the discovery of IR radiation in 1800. It has wide usage in both commercial and military applications. The IR spectrum can be divided into short-wave IR (SWIR) (1 to 3  $\mu\text{m}$ ), medium-wave IR (MWIR) (3 to 5  $\mu\text{m}$ ), long-wave IR (LWIR) (8 to 12  $\mu\text{m}$ ), and very-long-wave IR (VLWIR) ( $> 12 \mu\text{m}$ ). Among the cooled IR detector systems, PtSi and InSb can be operated only in MWIR. Si:As has a wide band spectrum from 0.8 to 30  $\mu\text{m}$ , but it can be operated only at temperatures around 12 K. Both HgCdTe (MCT) detector and quantum well infrared photodetectors (QWIP) offer wavelength flexibility in MWIR, LWIR, and VLWIR.

MCT is the most extensively investigated semiconductor alloy system for IR detectors. During more than 30 years of research, significant progress has been made in MCT materials, growth, processing, passivation, substrates, and manufacturing capability. Large format IR focal plane arrays (FPAs) have been demonstrated with pixel format up to  $1024 \times 1024^3$  for SWIR, and  $640 \times 480$  for LWIR. A  $128 \times 128$  array at VLWIR (15  $\mu\text{m}$ ) has been demonstrated by Rockwell using planar structure grown by molecular beam epitaxy (MBE) [1]. QWIP is a relatively new technology that has progressed very quickly in the past 10 years [2]. N-type GaAs/AlGaAs and InGaAs/AlGaAs systems on GaAs substrates are the maturest systems. Most of the emphasis in QWIP development has been on the LWIR, and FPAs with up to  $640 \times 480$  pixels have been demonstrated [3]. At VLWIR (15  $\mu\text{m}$ ) a  $128 \times 128$  FPA has been demonstrated by JPL [4].

FPAs at 12-18  $\mu\text{m}$  are very useful in detecting cold objects such as ballistic missiles in midcourse. VLWIR sensors are very important in strategic missile defenses and space applications. In general, the longer the detection wavelength, the lower the operating temperature of an IR photon detector, such as MCT and QWIP. At VLWIR, the band gap of the MCT is quite narrow and impurity defects dominate the detector noise mechanism. The variation of  $x$  across the  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  wafer can cause a much larger spectral nonuniformity in VLWIR than in MWIR. For example, at 77 K, a variation of  $\Delta x = 0.2\%$  gives a cutoff wavelength variation of  $\Delta\lambda_c = 0.063 \mu\text{m}$  at MWIR ( $\lambda_c = 5 \mu\text{m}$ ), while the same  $\Delta x$  can cause cutoff wavelength variations of  $\Delta\lambda_c = 0.25 \mu\text{m}$  for LWIR (10  $\mu\text{m}$ ), and  $\Delta\lambda_c = 0.5 \mu\text{m}$  for VLWIR (14  $\mu\text{m}$ ). Therefore, the required composition control on MCT is much more stringent for LWIR and VLWIR than for MWIR. For all these reasons, it is very

difficult to develop large format, uniform VLWIR MCT FPAs operating at temperature higher than 77 K [5].

QWIPs use intersubband transition instead of direct interband transition. III-V materials are used, and these have a relatively wide band gap (1.43 eV for GaAs). The advantages of a wider band gap material are that it has superior bond strength and material stability, well-behaved dopants, thermal stability, and intrinsic radiation hardness. Large and high quality GaAs substrates and mature GaAs growth and processing technology guarantee highly uniform large area FPAs with well-controlled molar compositions. The hardness of the material and substrate make device processing and array fabrication easy to handle, which leads to a high yields for the FPAs. The disadvantage of QWIPs is that the energy band gap does not fall in the IR regime, and thus a direct band gap transition cannot be used for IR detection. The intersubband transition sets certain fundamental limits on the device performance at  $T > 80$  K. When extends to VLWIR, the operating temperature of a QWIP drops to around 40 K at 15  $\mu\text{m}$  cutoff wavelength [5].

During the past few years, uncooled infrared (IR) sensors have been rapidly developed into large format FPAs. Impressive progress has been made in both resistive microbolometers and pyroelectric thin-film detectors with noise equivalent temperature differences (NETD) projected to be 10 to 20 mK with F/1 optics. The most mature uncooled IR detector arrays use thin film structures to reduce the thermal capacitance. The absorption of these detectors can be increased by constructive resonance conditions. To reduce thermal conductance, thin film structures possessing freestanding air bridges are often used in today's uncooled detectors. The advanced microelectromechanical (MEM) technology has made such detector structures possible. The advantages of uncooled IR thermal detectors are that they are intrinsically wavelength-independent, small size, lightweight, low cost and low power consumption. However, the sensitivity of the uncooled IR detectors are usually lower than that of cooled IR detectors, therefore there are many difficulties to suit uncooled IR detectors for DoD-related applications.

High- $T_c$  superconducting (HTS) bolometric IR detectors offer the promise of matching the sensitivity of MCT detectors, but with a detection range extended to LWIR and VLWIR. Above 15  $\mu\text{m}$ , the sensitivity of MCT at 77 K is severely limited by thermally generated dark current. In contrast, HTS bolometers have a much flatter power spectral response. Using HTSs, such as  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (YBCO), for microbolometer has been studied before and free standing air bridge structure has been demonstrated using YBCO with YSZ as a supporting layer [6-8]. The high detectivity ( $D^*$ ) of  $8 \times 10^9 \text{ cm Hz}^{0.5} \text{ W}^{-1}$  achieved on a single  $50 \times 50 \mu\text{m}^2$  pixel-area YBCO bolometer at 77 K and 32 Hz with detection wavelength larger than 15  $\mu\text{m}$  [6], approaching the sensitivity of MCT detectors ( $D^* = 2 \times 10^{10} \text{ cm Hz}^{0.5} \text{ W}^{-1}$ ). Further improvement of HTS film quality, such as the film epitaxy [6] and surface morphology [9], would yield a  $D^*$  comparable with that of MCT but in the LWIR and VLWIR range, displaying a great potential of the HTSs for LWIR and VLWIR IR detection. Furthermore, the operation temperature of the HTS bolometric IR detectors can be increased to above 130 K using higher- $T_c$  Hg-based superconducting (Hg-HTSs) thin films [10,11]. This makes battery operation of HTS bolometric IR detector possible using the state-of-arts thermoelectric cryocoolers. As the HTS film quality advances, the ultimate limiting factor for the HTS bolometric IR detector is mainly the phonon noise, which can be minimized by employing a suspended structure to improve thermal isolation of the detector structure [8]. Finally, with the

design of a resonant cavity between the ROIC and the detector structure, the IR absorption efficiency of the detector can be raised up to 80% [17].

## 1.2 Merit of Science

A HTS bolometric IR detector has a multilayer structure including generally layers for electrode, HTS, support, and sacrificial which will be removed later to form a suspended bolometer. A key issue is the growth of epitaxial HTS film on un-oriented sacrificial (such as polyimide) layer. Considering integration with si-ROIC, the HTS bolometer should be grown on si-ROIC, which implies a low thermal-processing budget below 600 °C. For IR imaging, focal plane arrays are necessary. This requires large-area uniformity (> 2inch in diameter), smooth surface morphology ( $R_a < 5$  nm), high growth rate, and preferably in situ growth of the bolometer multilayers in order to achieve a high product throughput. Finally, the compatibility of commercialization requires high throughput, high sample reproducibility, low capital investment and low running cost. Such requirements exclude most existing thin film fabrication techniques.

In order to obtain an epitaxial layer on polycrystalline or amorphous substrates, ion-beam-assisted deposition (IBAD) has been recently adopted and promising results were achieved [12]. In the IBAD process, a low-energy ion beam is directed on the sample during the growth so that preferable orientations can be induced in the film grown on polycrystalline or amorphous metal substrates. Moreover, other beneficial effects, such as reduced growth temperature, higher film density, smoother surface morphology, and improved electric properties, have also been observed. Since the ion beam requires high vacuum (better than  $10^{-5}$  Torr), it cannot, however, be incorporated directly into systems operated at low vacuums, which is unfortunately the case for most thin film deposition techniques for ceramics, including pulsed laser deposition, magnetron sputtering, and chemical vapor deposition. Instead, a second ion source is used for deposition, which results in an extremely low deposition rate (~ 1 nm/min.) and therefore is not suitable for industrial processing, which requires large-scale high throughput capabilities.

Thermal evaporation technique has long been demonstrated to be a simple and effective way to coat metals in large areas with high speed and has been widely used in industry. It uses simple metal as the source and works in the same vacuum range above  $10^{-5}$  Torr as the ion beam source. Since most perovskite materials and oxide electrodes are compounds, especially complex oxides involving more than one elements, the thermal evaporation method was not considered to be appropriate due to the requirement for high processing oxygen pressure and fine control of the composition. A recent development of the co-evaporation technique using a differential oxygen partial pressure produces high-quality Y-Ba-Cu-O films up to 9 inch in diameter with high growth rate of 24 nm per minute [13,14]. Combined with the simplicity of the material source selection and low capital investments of the system, the thermal co-evaporation technique is superior to other existing thin-film growth techniques for commercialization of oxide-based devices [15]. The limitation of this co-evaporation technique is that the substrate has to be single crystal in order to grow epitaxial films on top of it.

In the proposed work, an ion-beam assisted thermal co-evaporation (IBATCE) technique will be developed, that allows fast and large-area growth of epitaxial thin films and multilayers on a polycrystalline/amorphous substrate. By combining the merits of two state-of-the-art/industrial

proven technologies, the IBATCE provides several advantages: (1) growth of epitaxial or crystal oriented films on polycrystalline/amorphous structures, such as polyimide, metal contacts. (2) Allowing low-cost, large-area, etchable, lattice-mismatched, polycrystalline/amorphous substrates to replace relatively expensive, smaller area, and unetchable oxides substrates, such as SrTiO<sub>3</sub> and LaAlO<sub>3</sub> currently used for HTS bolometers. (3) High growth rate and large-area uniformity; (4) reduced growth temperature; (5) high film intensity and better surface/interface morphology, and (6) in situ growth of most metals and oxides.

### **1.3 Collaboration with other institutions**

The proposed research will be carried out in extensive collaboration with US Army research laboratory (ARL) on development of IBATCE technique, HTS multilayer structures for IR FPAs, and prototype devices. Many other institutions, such as Texas Center for Superconductivity (TCSUH) and Sandia National Laboratory (SNL), will also be involved in on thin film multilayers characterization and device processing. The expertise and the state-of-arts facilities are an important factor to the success of this project.

### **1.4 Relevance to DOD's Interests**

IR detectors and FPAs are very important to many DoD applications, such as night vision, surveillance, security, missile tracking, guiding, and intercepting. For example, ballistic missile defense scenario expects emerging threats to have colder targets, decreased object spacing, greater use of decoys and countermeasures, and longer-range detection compared to the current tactical scenarios. According to these ballistic missile defense requirements, IR FPAs need to be developed at VLWIR that have high sensitivity, large format, high uniformity, high operability, and relatively low cost and low power consumption. HTS bolometers offer solutions to the above needs with a low cost microbolometer structure, long detection wavelength (> 15 μm) and operating at relative low temperatures ~ 80-120K.

### **1.5 Impact**

The impact of IBATC will be tremendous on manufacturing high performance oxide-material-based devices using, such as superconductor, ferroelectric, paraelectric, oxide electrodes, and colossal magneto-resistive materials, which promise of a new generation of advanced high-performance microelectronics devices [15]. The applications are widely spread in, for example, IR detectors, memory, microwave, telecommunication and superconducting quantum interference devices (SQUIDs). Such devices may transform various technologies, potentially creating new multibillion-dollar markets. On the other hand, the proposed research will provide excellent opportunities to the graduate students to work on the advanced sciences and technologies through an extensive interaction with experts in industry and national laboratories. The ultimate goal of this project is to initiate a competitive project in Kansas on the emerging oxide-material research and to bring in significant amount of federal research dollars.

## 2. Proposed research

### 2.1 Description of the Equipment

The ion-beam assisted thermal co-evaporation (IBATC) technique employs the IBAD in the thermal co-evaporation process. As shown in Fig. 1, the sources are heated to their melting temperatures and the mixed source vapors are transport to the substrate for growth of metal or oxide thin films. The substrate can be heated with an infrared heating bank in the growth chamber and can be exposed to oxygen or other gases in the gas compartment. The gas pressure in the growth chamber and the gas compartment may differ by several order of magnitudes, allowing high vacuum to be maintained in the growth chamber. An ion gun is directed at a certain angle (preferably at near 45 degree) with respect to the normal of the substrate. An ion beam (such as argon or oxygen, or the mixture) bombards the substrate at an energy (typically a few hundreds eV) constantly during the growth to assist the crystal orientation formation of the sample. Another one or two ion guns are optional to either aim at the sample for breaking the chemical bonds which can reduce the substrate temperature during growth, or at the target for IBAD to grow a template layer before thermal evaporation, or both.

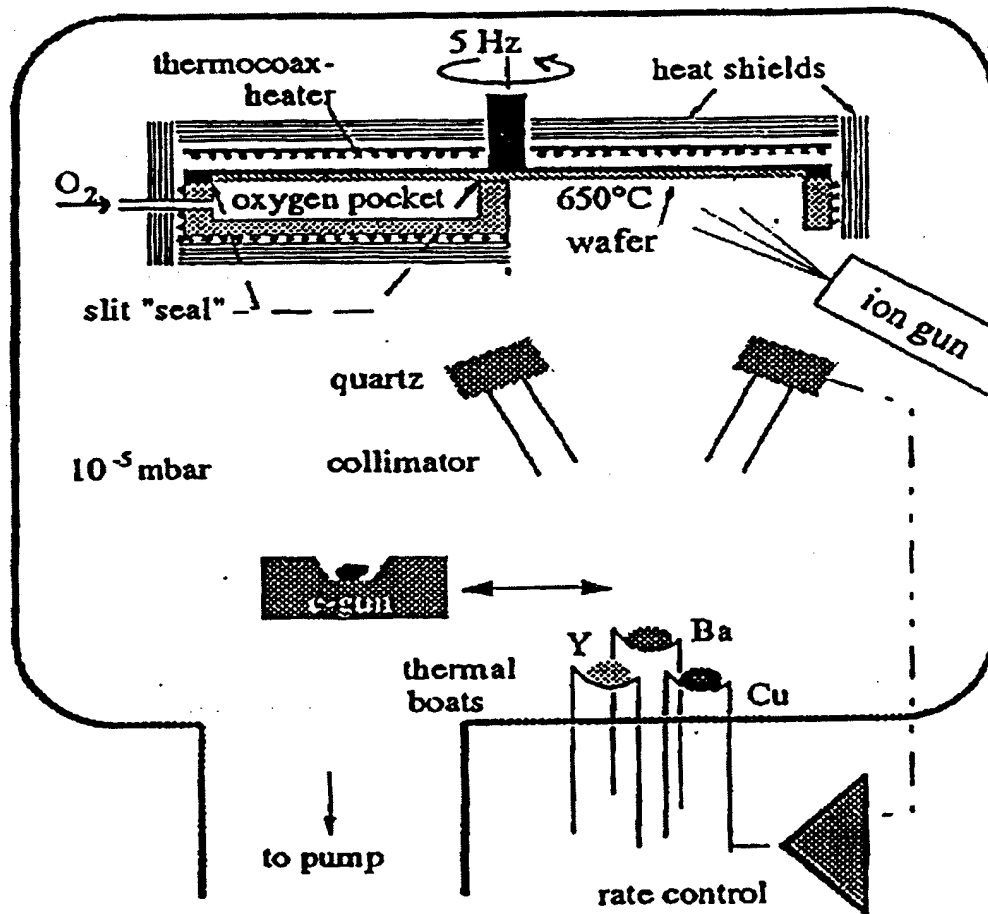


Figure 1. Schematic description of an IBATCE system.

An IBATCE system to be constructed is composed of six major parts: vacuum chamber, differential pumping station, material source evaporation, substrate heating, Ion-beam assistance, and *in situ* monitoring and controlling. During the sample growth, the pumping system maintains a high vacuum in the main chamber, where the source evaporation transportation occur, and a low vacuum in the oxygen chamber, which is connected through a narrow gap (< 1mm wide) with the main chamber. The sample can be transferred between the main and oxygen chamber via this gap during the deposition enabling oxidation of deposited metals to form a HTS or other oxides. The sources of various simple metals and a large variety of compounds are thermally evaporated and the vapors of the material mixture are transferred diffusively to the substrate heated, if necessary, with an IR heating bank. A shutter near each source switches on/off transfer of this source, enabling growth of multilayers of different materials. A beam of Argon or Argon and oxygen ions with their energy above 500 eV is directed at the sample during growth of HTS mechanisms, such as an ellipsometer, will be employed to *in situ* monitor the thickness, composition and interface roughness of the sample.

## 2.2 Material Issues in HTS bolometer Heterostructures

Fig. 2 is a resonant cavity structure similar to those used in uncooled IR detectors [16]. We use it to depict one of the multilayer HTS structures to be fabricated using the IBATCE system. The growth sequence on the si substrate consists of: (1) thermal evaporation of a sacrificial layer of polycrystalline rock salts; (2) IBATCE of the supporting layer of YSZ; (3) thermal evaporation of metal electrode; and (4) IBATCE of HTSs.

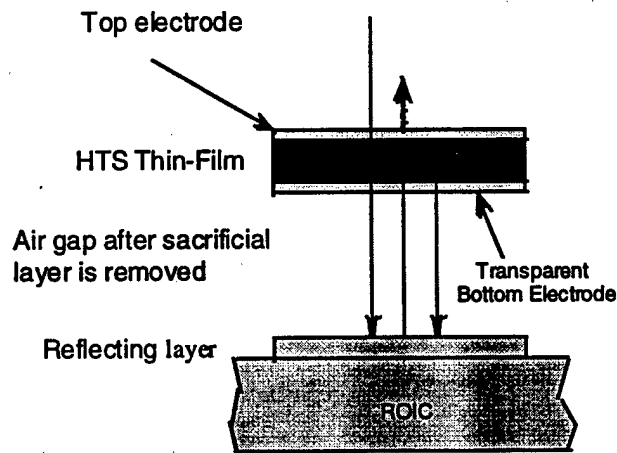


Figure 2. Cross-sectional view of a HTS bolometer with resonance cavity.

**Substrate** HTS bolometers need to be integrated with Si-ROIC. This requests growth of HTS bolometer heterostructures on si substrates, which are inexpensive and available in large size.

**Sacrificial layer** will be removed after the device processing to form air-filled gap between the HTS bolometer and the Si-ROIC. By effectively reducing the thermal conductance, the sensitivity



of the IR detector can be significantly improved. Considering the problems in the currently adopted sacrificial layer-polyimide, we will use rock salts, such as LiF and NaCl as the sacrificial layers.

Supporting layer is used as the template for growth of high-quality epitaxial Ht. on polycrystalline sacrificial layer. YSZ has been well proved for such a purpose in the previous study.

Electrodes can be selected from simple metals, such as metals and metallic oxides including  $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$  and  $\text{CaRuO}_3$  using IBATCE technique. The large variety of the electrode selection is advantageous to the device design.

HTSs will be used to fabricate bridge-type or meandering-line type bolometer arrays for IR detection. Both  $\text{YBa}_2\text{Cu}_3\text{O}_7$  and Hg-based HTSs will be considered. The former has been studied extensively for bolometer applications with performance close to that of MCT IR detectors at 77K. A better performance is expected as the quality of the film, such as the surface/interface morphology is improved and device structure optimized. On the other hand, Hg-based HTSs have the highest  $T_c$  near 135K, which offers opportunities of device operation up to 130K and possibilities to be integrated with the thermal-electric cryocoolers.

The focus of this research is on the growth mechanism of the various layers in the IBATCE process. The processing conditions will be correlated with sample characterization using XRD, SEM, TEM, AFM, and electric transport measurements. A prototype HTS bolometer IR detector will be fabricated and the processing parameters will be optimized with respect to the performance of the device.

Compatibility of a HTS bolometer FPA with the Si-ROIC is a basic requirement for commercialization. This sets a strict upper limit of 600 °C for the thermal budget of the HTS bolometer, which is 150-200 °C lower than that used to grow HTS thin at fairly high oxygen partial pressure (>100 mTorr). In order to solve this problem, an ion-beam-assisted growth at very low oxygen partial pressure will be employed in this research. According to the chemical phase diagram, HTSs such as YBCO can be formed at lower temperature at lower oxygen partial pressure, while the processing window becomes narrower.  $\text{NO}_2$  will be used to provide atomic oxygen, which promotes oxidation of metals at much lower temperature near 600 °C. On the other hand, the ion-beam energy will be adjusted to provide, besides the in-plane alignment of the HTS grain, extra energies to reduce the thermal budget.

Meanwhile, film transfer method may also be a viable way to integrate the HTS bolometer FPR with Is-ROIC. In this case, the HTS bolometers FPAs grown on Is-substrates will be simply pressed on the si-ROIC after the sacrificial layer is removed.

### 2.3 Device Fabrication and Characterization

Suspended HTS bolometer will be fabricated and Fig. 3 shows the schematic of this device. The pixel size will be 50-100  $\mu\text{m}^2$ . Several considerations will be given to improve the performance of the HTS bolometer by minimize the noise level.

Temperature sensitive  
transducer material (HTSs)

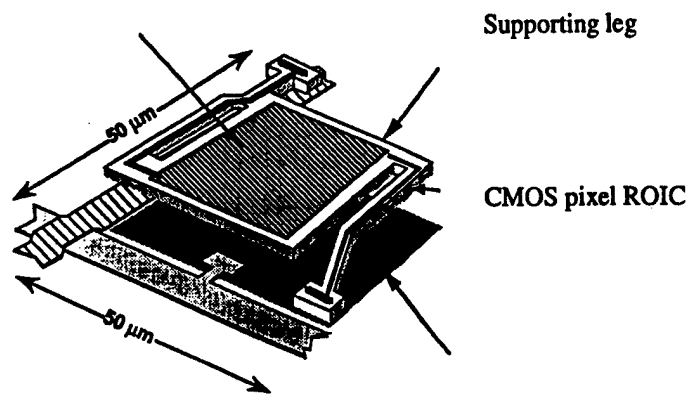


Figure 3. Single pixel of suspended HTS bolometric IR detector [16].

Generally, there are four types of noises, which could degrade the performance, such as the detectivity ( $D^*$ ). They include  $1/f$  noise, phonon noise, Johnson noise, and amplifier noise. The first two are the dominant factors for HTS bolometer. The HTS film due to, for example, poor superconducting properties and poor film epitaxy and surface morphology generates the  $1/f$  noise. For poor-quality HTS films, the  $1/f$  noise dominates the phonon noise so that it is of first priority to achieve high quality epitaxial HTS thin films with smooth surface morphology. The high-quality YBCO thin films, the  $1/f$  noise is reported to be less than  $1 \text{ nV/Hz}^{0.5}$ , at which the phonon noise becomes the major limiting factor. The phonon noise is proportional to the thermal conductance of the bolometer. Employing the suspended structure as shown in Fig. 3 can thus reduce the phonon noise. The proposed IBATCE technique aims at to grow high-quality HTS bolometers. To optimize the bolometer performance, emphasis will be given to improving the film quality and to minimizing the thermal conduction.

### 3. Research Plan

The ultimate goal of the proposed research is to develop an industrial viable technique for fabrication of HTS bolometer IR FPAs with approaching  $D^* > 2 \times 10^{10} \text{ cm Hz}^{0.5} \text{ W}^{-1}$  in the LWIR and VLWIR ranges. In the following table, the milestone for each year during the three-year project period is listed:

year	objectives	Goal
First	Design, construction and testing of the IBATCE system	IBATCE fully operational
Second	Growth of HTS bolometer heterostructures and various characterization	Resolving material issues and optimizing growth process
Third	Fabrication of single pixel suspended HTS bolometer and device characterization	$D^* > 2 \times 10^{10} \text{ cm Hz}^{0.5} \text{ W}^{-1}$ at 32 Hz frame frequency

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