REALIZATION OF A LOW-COST, SPECTROSCOPIC, MID-INFRARED IMAGING PLATFORM FOR EXPLOSIVES DETECTION AND THERMAL TIME-HARMONIC IMAGING

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by David J. Benirschke

Scott S. Howard, Director

Graduate Program in Electrical Engineering Notre Dame, Indiana September 2019

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Abstract

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This report is split into two parts the first part outlines the development of a low-cost, spectroscopic, mid-infrared imaging platform for enabling applications. Particular motivation is taken from the problem of detecting explosives at security checkpoints. The second part looks at time-harmonic (phasor) based thermal imaging. The theory is first developed both analytically and numerically. This is followed by experimental verification. Further elaboration is done in the following two sections.

Explosives detection

Trace explosives detection has long been sought by security administrations worldwide. Due to this need, much research has gone into the development of fast, discreet, and sensitive trace detection methods. The most successful and effective technology today is that of chemical swabs paired with mass spectroscopy. As this technique is relatively time consuming it cannot feasibly be performed on every object or person entering the checkpoint forcing security agents to resort to random sampling. In addition, this technology could never be made into a stand-off configuration and will always put the operator in potential danger. In order to overcome these drawbacks optical detection methods have been developed. One of the most promising of these optical techniques is long-wave infrared (LWIR) spectroscopy. However, LWIR systems have not yet been implemented at the checkpoint due to both their cost and current detection limitations. This report outlines research and development towards the realization of a low-cost alternative to the current LWIR detection platforms alongside exceeding the current detection limits.

The first step towards developing any spectroscopic imaging platform is the identification of the wavenumber (or equivalently wavelength) range of interest. FTIR spectroscopy is used to study the spectra of the RDX comprised explosives C4 and Semtex. From this study a common spectral feature at 1260 cm^{-1} is discovered. In order to perform detection this spectral feature must be able to be seen in the presence of interferents and spectral shifts due to measurement condition or substrate variations. To do so the chemometric techniques of principal component analysis (PCA), second-derivative spectroscopy, and Savitzky-Golay (SG) filtering are applied to the trace detection problem. An experiment is conducted to show that these techniques can be used to positively discriminate Semtex from a plastic substrate. To speed up the application of these procedures, easy to use MATLAB GUI tools are developed.

After studying the spectral features of explosives, the report turns to low-cost LWIR detector alternatives, specifically the vanadium oxide (VOx) microbolometer. Many LWIR spectroscopic imaging applications are currently limited by the portability and cost of detector arrays. Recently, however, there has been an influx of low-cost, commercially available VOx LWIR detectors that are designed for non-scientific applications. Due to their intended market, these devices have not been thoroughly characterized. In an effort to alleviate some of the cost of trace detection systems the system performances of these low-cost VOx devices are investigated. The system characteristics are discussed in terms of the camera responsivity spectrum, noise equivalent differential temperature (NEDT), and Allan variance. Results of this analysis show an

optical response between $\lambda = 7.4$ to 12 μ m, noise equivalent differential temperature (NEDT) of 140 mK (at ≈ 7 fps), and demonstrated hyperspectral imaging of SiN thin films. Compared to a video frame rate, research-grade camera, these low-cost cameras achieve the same sensitivity with a 93x longer integration time and a 4x and 48x reduction in weight and cost, respectively. While this is a large relative discrepancy, for measurement sensitivities of interest the current integration time for the VOx microbolometer is 1.5 seconds.

With the camera properly characterized, attention is turned to the development of the trace detection platform. First, high level system design is presented using a tunable quantum cascade laser (QCL) as the source and a commercially available, phone-adapted VOx microbolometer as the detector. During the development of the data acquisition software it is discovered that the camera has multiple operational modes. The mode nominally used by one's phone has a frame rate of 7 Hz. However, with minimal hardware and software modifications the camera can be ran at a framerate of 32Hz. This greatly improves the viability of using this camera as a detector and improves the above reported trade-off to only a 20.43x longer integration time. After the development of the physical system the data acquisition, GUI design, and a client-server methodology for stand-off applications is outlined. With the hardware and software completed a low-cost trace explosives detection platform has come to fruition.

Initial measurements are presented showing the current measurement capabilities of the detection system and difficulties arising from the coherent imaging phenomena speckling. After the initial measurements, future work is proposed towards the testing and improvement of the Receiver Operator Characteristics (ROC) of the current system. To do so a collaboration with Professor Gupta's group has been established to develop optimal sensor selection algorithms. In order to do so a multitude of different measurement modalities must be developed. The report concludes with the description of the different measurement modalities to be investigated, including reflection spectroscopy, angle-resolved reflection spectroscopy, thermal life-time imaging, and polarization imaging.

Time-harmonic (phasor) thermal imaging

In the second part, phasor based thermal imaging is investigated. It is shown that the thermal value \sqrt{D}/k can be measure from the front side of the surface. Where D is the diffusivity and k the thermal conductance. It is important to note that the common laser-flash diffusivity measurement requires viewing access to the back side of the sample of interest, but it can isolate the diffusivity and conductivity. However, for many imaging applications this composite value is perfectly acceptable and becomes a way to discriminate substances or interrogate its local surroundings.

To develop this theory, first, the phasor solution for the homogenous heat equation is derived alongside a numerical method to compare results. Excellent agreement is found between the numerical method and analytical methods. Next, the numerical method is extended to two materials in contact with one another. This shows, surprisingly, that the surface phasor amplitude (temperature) can be enhanced in the two material case relative to the first case by choice of certain materials (and equivalent diffusivities and conductivities). Next, experiments are done that show excellent agreement between theory and experimental results. Finally, future work is proposed to develop this theory into a full-fledged theory thermal diffuse tomography.

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PART I:

REALIZATION OF A LOW-COST VANADIUM OXIDE BASED SPECTROSCOPIC IMAGING PLATFORM FOR EXPLOSIVES DETECTION

CHAPTER 1

INTRODUCTION AND MOTIVATION

1.1 Explosives detection

Fast, discreet, and sensitive trace explosives detection at the checkpoint (e.g. airports or train stations) has long been desired by defense agencies worldwide. The most effective, currently implemented explosives detection technology is physical sampling and detection, e.g. swabbing paired with mass spectroscopy [18]. Swabbing is accomplished by having an operator rub a swab across the surface being interrogated. These swabs have been engineered specifically to collect trace explosives and have them adhere to the surface. This swab is then placed into a mass spectrometer which determines if explosives residues are present. |41, 46|. While swabbing boasts incredibly high sensitivity it requires an operator to come in contact with the object of interest putting them at risk while also not being discreet. Additionally, due to the time required for the operator to obtain the sample and process it in a mass spectrometer, swabbing can not feasibly be performed on all people and baggage in an area requiring high throughput. This speed limitation forces checkpoint operators to resort to random sampling of passengers and their luggage. If truly random sampling is done it is overwhelmingly likely that anyone attempting to bypass security will in fact get through. This is simply due to the fact that the number of threats are a massive minority in comparison to the total number of passengers. This deficiency was demonstrated by a Department of Homeland Security (DHS) undercover test of the Transportation Security Administration's (TSA's) security at airports, where

swabbing is used. While the exact data of this test is confidential, news agencies reported the failure rate at around 80%. Since the validity of mass spectrometry for explosives detection has been definitively shown, this failure must fall to the operators and random sampling [17]. As the number of passengers continues to increase each year the probability of detecting a threat with random sampling continues to decrease. This is ultimately a fatal limitation of swabbing and alternative or complementary techniques must be developed.

To combat the deficiencies of swabbing, researchers have developed optical detection methods, primarily that of long-wave infrared (LWIR) spectroscopy [6, 59, 19]. In contrast to swabbing, LWIR systems can be designed to be stand-off, discreet, and extremely fast; feasibly allowing for all people and baggage to be scanned. A visualization of how optical-based explosives detection systems could fit into the checkpoint is shown in Figure 1.1 [59]. There are, however, a number of technical and financial difficulties that have kept LWIR methods out of the checkpoint that remain to be solved adequately. The research presented in this report is focused on alleviating some of the financial burden of LWIR trace detection platforms by utilizing alternative lower cost detection equipment alongside developing novel methods to overcome the current technical limitations. In order to understand the novelty behind these developments an introduction to the science and technology behind LWIR spectroscopic imaging is required, and is done presently.

1.1.1 Long-wave infrared absorption spectroscopy (LWIR)

As with all optical spectroscopic techniques, LWIR spectroscopy looks to measure an interaction between electromagnetic radiation (EMR) and the sample of interest. The interaction for which we are interested is single-photon absorption as described macroscopically via the Beer-Lambert law. The Beer-Lambert law predicts that the optical power will decrease exponentially as it propagates through a material. This



Figure 1.1. Visualization of a potential checkpoint utilizing optical detection methods [59].

decay is characterized by the absorption parameter α such that the total power after propagating a distance x through a material is $I(x) = I_0 e^{-\alpha x}$, where I_0 is the incident power. α is a property of the material being interrogated and is in general a very strong function of wavelength. Plots of α versus wavelength (or any other equivalent parameter e.g. wavenumber, energy, etc.) is called a spectra.

In LWIR spectroscopy we are using light residing in a particular region of the mid-infrared (MIR). The MIR region of the EMR spectrum is bounded from below (on a scale of wavelength (λ)) by the red part of the visible (0.7 μ m) and from above by the terahertz (100 μ m). It is important to note that it is customary to use wavenumbers instead of wavelength when discussing LWIR spectroscopy. Wavenumbers are defined as the reciprocal of the wavelengths and are reported in units of cm^{-1} , i.e. $\sigma(cm^{-1}) = 10^4/\lambda(\mu m)$ where σ is the wavenumber. Therefore, the MIR region spans the wavenumber range from 100 cm^{-1} (100 μ m) to 14286 cm^{-1} (0.7 μ m). As the wavenumber of EMR increases (or equivalently wavelength decreases) the corresponding photon energy increases. Due to this proportionality, MIR photons do. Instead, when MIR photons are absorbed by a molecule they excite vibrational and rotational states of the molecule. These vibrational and rotational energies are characteristic of the bonds and constitutive elements of molecules, allowing for

identification of a molecule by its MIR spectrum. The region best suited for molecular identification is that from 500 cm^{-1} to 1500 $cm^{-1}(6.7 \ \mu m$ to 20 $\mu m)$. This region is designated as the molecular fingerprint region as a molecule's spectrum can be treated as a unique identifier, i.e. its "fingerprint". The higher energy spectral region between 1500 cm^{-1} and 14286 cm^{-1} (0.7 μ m to 6.7 μ m) is also used for molecular identification, but is only capable of identifying functional groups [8]. We will refer to this region as the functional group region. Differences in identification capabilities of these two regions can be seen in Figure 1.2. In this figure, spectra of the two isomers of propanol are shown side by side. Molecules are said to be isomers of one another if they share the same chemical formula, but have different chemical structure. Looking at the functional group region around and above 3000 cm^{-1} one observes the broad absorption peaks in both spectra that are present in all alcohols, but it hard to differentiate the two propanol by this alone. To do so one must look at the fingerprint region. In the fingerprint region there are many more, sharply defined absorption peaks. Comparing the two there are differences such as the number of peaks around 1000 cm^{-1} and the existent of a peak at 800 cm^{-1} in 2-propanol that is not present in n-propanol. However, one can immediately see that the fingerprint region is much harder to interpret than the functional group region due to its complexity. This has been a major difficulty in LWIR optical detection and will be further discussed in the coming chapters. It should be noted though that the fingerprint region is essential as most explosives are organic compounds. This means that they share the same functional groups with common substances such as oils and cosmetics. Using only the functional group region would thus leave explosives indiscernible from common everyday organic materials which are plentiful at checkpoint scenarios.



Figure 1.2. Spectra of the two isomers of propanol. Notice how the two spectra are very similar in the functional group regions, but differ in the fingerprint region.

In addition to being qualified for unique detection, the fingerprint region is also attractive for stand-off detection system as a significant portion of it resides within the atmospheric window from 714 cm^{-1} to 1250 cm^{-1} (8 μm to 14 μ m), allowing long pathlength propagation through the atmosphere with minimal attenuation and is eye safe up to very high powers, Figure 1.3. With this in mind we will now define the term LWIR radiation more precisely to be EMR within this atmospheric window, as is common in military applications.

1.1.2 LWIR spectroscopic imaging

Due to the advantages previously stated, LWIR optical spectroscopy has been found to be a powerful technique for sensitive and selective measurements of trace chemical compounds for defense, environmental sensing, industrial, and medical applications [6, 12, 4, 32]. In order to conduct LWIR spectroscopic experiments one needs to use both a source of LWIR EMR to illuminate the object of interest and a detector to "see" it.

High power LWIR sources were historically hard to come by, but the development of the quantum cascade laser (QCL) changed that drastically. The QCL has become



Figure 1.3. Transmission of EMR from 200 nm to 20 μ m as calculated by MODTRAN using the following model input: atmosphere, subarctic summer; aerosol, rural 23 km; and path, 1 km horizontal near the ground. Reproduced from [59]. The region of interest for the current application is the atmospheric window on the far right side of the figure lying between the

 H_20 and $C0_2$ broad absorption peaks.

the go to ubiquitous source for almost all LWIR chemical sensing applications. This is because QCLs can achieve narrow-bandwidth, widely tunable emission in the range of $\lambda \approx 4$ to $\lambda \approx 12 \ \mu m$ and continuous wave powers exceeding 2 W [61, 36]. While QCLs were originally prohibitively expensive (e.g., > \$50,000) and required a high level of technical expertise to operate, these devices are now commercially available from several vendors in convenient, widely tunable packages (Daylight Solutions, Inc.; Block Engineering, Inc.; and Pendar Technologies, LLC) as well as relatively low-cost single-wavelength mounted chips (ThorLabs, LLC, for < \$5,000).

The counterparts to LWIR sources, the detectors, are generally classified as either photon detectors or thermal detectors. In photon detectors, free-charge carriers are generated through the absorption of photons without significantly increasing detector temperature (e.g. P-N junction) [16]. Photon detectors are highly sensitive, but require significant cooling to reduce dark-current noise [16, 48]. Thermal detectors indirectly measure optical power by absorbing the radiation in order to heat the material, and subsequently measure a temperature dependent property of a material

[16]. For example, microbolometers are thermal detectors that use changes in electrical resistance. Thermal detectors are generally much smaller, less expensive, and require less cooling than photon detectors at the cost of lower sensitivity and a slower response [16, 48]. LWIR spectroscopy applications typically use highly-sensitive, but expensive, single-element photon detectors (e.g. mercury cadmium telluride (MCT) detectors) that require large amounts of power or liquid nitrogen to cool. Photon detector imaging arrays are available, but are even more expensive and bulky [49]. The cost and cooling requirements thus limit photon detectors to centralized laboratory environments. In order to enable field-deployable LWIR spectroscopic imaging, lowcost (and lower sensitivity) thermal imaging arrays have received attention from the LWIR community. Most common of these are microbolometers, making up 95% of the uncooled detector market in 2010 [49]. Microbolometers are temperature dependent resistors that absorb electromagnetic radiation, therefore irradiance can be inferred from measurements of microbolometer resistance. This optical power value is used to determine the temperature of the radiation source. Most IR microbolometers are made to absorb in the range of $\lambda\approx 8$ to $\lambda\approx 14~\mu{\rm m}$, corresponding to the second atmospheric transmission window [37]. Currently the most common material used for microbolometer fabrication is vanadium oxide (VOx). This is due to its high temperature coefficient of resistance (i.e. responsivity) and low 1/f noise characteristics [49, 31].

MIR focal plane arrays are used for two types of spectroscopic imaging: passive and active. In passive imaging, the detector images a target that is emitting MIR radiation. There is no illumination of the target by a controlled source. An example of this would be thermography which can be used to monitor chemical reactions or identify electrical faults [5, 24]. Active spectroscopic imaging systems illuminate the target with a controlled source of MIR radiation (e.g. a QCL). The image is then a combination of the target's natural MIR radiance and the reflected power of the illuminating source. Spectroscopic imaging is realized by either varying the source wavelength or by modulating each wavelength [30, 42].

1.1.3 Raman spectroscopy

It is appropriate to briefly discuss one non-absorption based MIR spectroscopic method due to its popularity, and this is Raman Spectroscopy. Raman spectroscopy does not measure the absorption of radiation, but rather an inelastic scattering process. Raman works by illuminating the sample with EMR typically ranging from the near UV to the near infrared. Three peaks will be present in the Raman spectra; one at the excitation wavelength (Rayleigh peak), and two peaks on either side offset by the same distance. The peak at higher energy is known as the anti-Stokes peak and the peak at lower energy is known as the Stokes peak [34, 8]. While Raman is incredibly sensitive it suffers from having low signal levels. Because of the disadvantages in cost, eye safe power, and low signal; Raman is not used in stand-off explosives detection and we will not be pursuing it further. However, it is important to note that trace explosives detecting hand scanners utilizing Raman have been developed and are commercially available [56, 11]. The down side to these devices being the long amount of time required to complete a measurement and that the scanner must be in almost direct contact with the object under interrogation.

1.1.4 Current research in explosives detection

Explosives detection has been an active area of research for a long time now and LWIR detection platforms have come to fruition. Most notably, incredible progress has been made by the The Fraunhofer Institute and U.S. Naval Research Laboratory [26, 27, 10]. While the systems these groups, and others, have developed are incredibly sensitive, they have not been implemented at the checkpoint due to both cost and lower than desired substance identification capabilities. The detection platforms realized so



Figure 1.4. Explosive detection system from The Fraunhofer Institute, Germany. (b) shows a zoomed in version with components labeled. Not shown is a LN² cooled MCT which lies in the telescope behind the visible camera (Vis-cam) [21].

far have relied on LN2 cooled MCT detectors and widely tunable QCL platforms. In order to generate images with MCT technology one has to either have an array of MCTs or develop a scanning system, both increasing the cost of the system drastically. In addition, the size, weight, and power (SWAP) overheard of these systems is very high and does not lend itself easily to non-laboratory environments. A system that has been realized is shown in Figure 1.4 [21].

The low success in substance identification arises not from system reliability and data interpretation, but from the variability in real world measurement conditions and sample morphology along with the presence of organic interferents [29, 19, 57]. It was shown previously in Figure 1.2 that the fingerprint region has many spectral features and can be difficult to interpret. In addition to this, it has been shown that MIR reflection spectra of explosives vary with substrate, particle size, and angle of incidence [43, 20]. To be able to reliably detect explosives amongst these variations, much of the current research in this field has switched from hardware design to detection algorithm development such as machine learning, linear mixing models, or principal component analysis (PCA).

1.1.5 Low-Cost imaging with a vanadium-oxide microbolometer

While previous work has demonstrated the feasibility of explosives detection systems, they have remained prohibitively expensive and/or difficult to implement. To help reduce the cost of these systems, low cost focal-plane arrays are necessary. Recently, VOx microbolometers have reached retail markets for consumer applications. Products include FLIR ONE (FLIR[®] Systems, Inc.) and SEEK Compact (Seek Thermal, Inc). These new products target residential thermal imaging applications at low cost (\approx \$250), well below that of any other commercially available microbolometer array (typically > \$1000). An example image from the SEEK Compact is shown in Fig. 1.5. These low cost, compact, LWIR sensitive detector arrays thus have a potential to enable field-deployable LWIR spectroscopic imaging for scientific applications. Referring again to Figure 1.1, it can be seen that the number of required detectors can grow very quickly depending on the area to be interrogated. For applications such as this to be implemented, it is imperative that the cost of an individual detection module be reduced. It is thus the goal of this research to attempt to lessen this cost by realizing a detection platform using a VOx microbolometer as a low-cost detection alternative. However, as these cameras were not designed for scientific research, they must first be characterized.

1.2 Overview of work

This report describes work developing, characterizing, and validating a low-cost spectroscopic imaging platform for the detection of trace explosive residues.

First, in Chapter 2 the spectral characterization of two RDX comprised explosives C4 and Semtex is conducted. It is shown that the commonly used chemometrics techniques of principal component analysis (PCA), Savitzky-Golay (SG) filtering, and second-derivative spectroscopy can be applied to explosive spectra as tools for the



Figure 1.5. Example image of the author's cat taken from the SEEK Compact used on an Android phone.

identification of RDX spectral features shared by the two explosives. Using these techniques the wavelength of interest for identification is chosen and used to select a QCL to be used as the LWIR detection source. Furthermore, GUI tools are designed and demonstrated to ease the application of these chemometric techniques.

Chapter 3 presents a thorough thermal imaging performance analysis of a representative low-cost VOx microbolometer array (Seek Compact, Seek Thermal, Inc). The thermal imaging specifications known as the noise equivalent differential temperature (NEDT), responsivity spectra, and Allan variance are presented to demonstrate the feasibility of low-cost VOx cameras for sensitive and quantitative spectroscopic imaging. Additionally, the results of a silicon nitride (SiN) thin film hyperspectral imaging experiment are presented to demonstrate imaging capabilities.

Chapter 4 is concerned with the design of a laboratory system for testing a VOx microbolometer as the camera of a trace detection system. In addition, software and hardware modifications are presented that have increased the framerate from 7 Hz to 32 Hz, greatly improving the imaging capabilities of the device. Afterwards, the development of a wireless system complete with easy to use GUI for potential stand-off configurations is developed.

Finally, in Chapter 5 the report is concluded with initial system measurements and

the proposition of future research geared towards improving system capabilities for the identification of explosive compounds utilizing different measurement modalities.

CHAPTER 2

EXPLOSIVES SPECTRA CHARACTERIZATION AND CHEMOMETRIC TECHNIQUES

In order to begin conducting explosives detection experiments, the spectra of the samples must first be known. While explosive spectra have been measured in the past, there has been large variations due to differing sample compositions. Therefore, it is important to first measure the specific samples to be used in testing the completed detection platform. After the spectra have been measured, ways of identifying common spectral features among various spectra are investigated. To do so three commonly used chemometrics techniques are employed: principal component analysis (PCA), Savitzky-Golay (SG) filtering, and second-derivative spectroscopy. Since these techniques are less known in the optics community a brief description is presented in the main text. Finally, the results of the application of these chemometric techniques to explosives spectral characterization is shown.

2.1 Explosives spectra characterization

To begin developing the LWIR imaging system, we first sought to optimize our system for peak sensitivity by performing LWIR spectroscopic imaging using an FTIR. Spectral analysis of the complete explosive (energetic material, binder, and plasticizer) yields the required laser spectra, tuning ranges, power spectral density, and imaging speeds for a required sensitivity and specificity. Trace samples (artificial fingerprints, see Figure 2.1) of C4 (91 % RDX) and Semtex-1A (76 % PETN, 4.6 % RDX) were deposited on plastic substrates. Samples were provided by ALERT thrust leader

Professor Steve Beaudoin at Purdue University. While the highly-explosive material (HEM) components of C4 and Semtex (PETN and RDX) are commonly characterized in the MIR [57] on similar substrates [43], and C4 is well characterized in laboratory settings [60], we sought to establish performance parameters of composite explosives on realistic substrates.

The experiment was set up so that the sample was illuminated at 45° by the FTIR and collected onto the LN₂ cooled MCT detector. Alignment was done by maximizing the output of the analog-to-digital converter (ADC) from the MCT. The incoming beam from the FTIR was focused by a 1.5" lens to an elliptical spot with a major axis length of \approx 450 µm and minor axis length of \approx 350 µm.



Figure 2.1. Fingerprint pattern of the explosive Semtex 1A on a plastic substrate. The fingerprint is the ellipsoidal white pattern in the center.

Since the location of the explosive on a sample can only be hypothesized by visual inspection and the MCT setup can only measure the spectra at a single point, multiple spectra were generated across the surface of a sample. A representative collection of spectra is shown in Figure 2.2 for Semtex 1A. Each curve on the plot represents a different physical measuring location on the sample of interest. All of the curves have been normalized to the largest peak at 1260 cm^{-1} . This peak has been verified by the literature to to be RDX [60, 51]. The peaks at 1096 cm^{-1} and 1016 cm^{-1} , however, have not appeared in all RDX spectra observed. We have thus attributed them to the plasticizing agent used in our particular composition of Semtex 1A. It is noticeable that the presence and height of the peaks changes drastically across different points on the sample. This variation is due to changing surface loading concentrations.



Figure 2.2. Various spectra of plastic with or without Semtex. The peaks at 1260 are due to RDX in the Semtex. It is believed that the peaks at 1096, and 1016 cm^{-1} arise from the plasticizing agent.

2.2 Chemometric techniques

2.2.1 Principal component analysis (PCA)

As stated before we are most interested in the molecular fingerprint region of the MIR. However, this region is typically very hard to interpret due to its rapidly varying nature between samples and differing measurement conditions. In addition since we are not isolating individual molecules, many of the spectral features observed do not correspond to the substance of interest. For example, the spectra of RDX will be simultaneously measured with that of the substrate, binder, and interferents. In order to properly identify the explosives one needs a means of identifying the relevant spectral features. Following much of the literature in explosives detection, the approach taken here is to use PCA. The following explanation of PCA follows from [28].

PCA takes the original data set and projects it onto a basis set with the condition that each subsequent basis vector, the principal components (PC), accounts for the most variance in the data while remaining uncorrelated with all other components. Let x be a data matrix with rows corresponding to measurements and columns to variables, z_k the to be determined k^{th} PC, and w_k the k^{th} column weighting vector. Then PCA can be mathematically expressed as,

$$z_{k} = w_{k}^{T} x : \begin{cases} maxarg(var(z_{k})) \\ ||w_{k}||=1 \\ cov(z_{k}, z_{l}) = 0, k \neq l \end{cases}$$
(2.1)

where ||w|| is the euclidean norm, $\sqrt{w_1^2 + w_2^2 + w_3^2 + \dots}$, $cov(z_k, z_l)$ the covariance between z_k and z_l , and $var(z_k)$ the variance of z_k . A maximizing condition must be chosen as one could increase the magnitude of the weighting coefficients without bound to maximize the variance. This optimization problem can be solved through the method of Lagrangian multipliers and doing so leads to the eigenvalue problem of,

$$(\Sigma - \lambda_k I)w_k = 0 \tag{2.2}$$

where Σ is the either known or estimated covariance matrix of x, λ is the Lagrangian multiplier, and I is the identity matrix. Thus the weighting vectors are the eigenvectors of the covariance matrix and the multipliers the eigenvalues. The largest eigenvalue

corresponds to the eigenvector that maximizes $var(z_k = w_k^T x)$, and thus the first PC. One can now sort the measurements into classes based on their coordinates in the new basis set. In the case of interest these classes would be if RDX is present or not.

The benefit of applying PCA is that it accomplishes dimensionality reduction. As an example consider the measurement of two variables x and y such that y = x. For the line y = x one can easily see that knowledge of one variable provides complete knowledge of the other variable. This suggests that we could simply describe this line with a single variable. Now lets assume we can measure x exactly but have noise in y and we wish to find the PCs that describe this data set. Two instances of these variables with varying noise levels in y are shown in Figure 2.3 along with the PC vectors. The legend insets are what percentage that a PC describes the variance in the data. For the low noise case in 2.3a it can be seen that the data is easily described by only PC1 as it accounts for 99.8 % of the variance in the data. The unit vector in the direction of PC1 is (0.7062, 0.7080) which is extremely close to a 45° angle from the x axis, as is expected. A data point's location along the PC1 axis can thus be considered to contain all information in the data set and reduces the set from 2-D to 1-D. In 2.3b it can be seen that the more noise that is added the more variance is attributed to the second PC. Therefore, there is loss of information in 2.3b if one considers only the first PC1, but there is essentially no loss in 2.3a.

The concepts of the 2-D case generalize easily to the N-D case. For spectra, the measured power at each wavenumber is a variable, making the dimensionality of our data space very large. In Figure 2.4a the spectra measured at different locations on a sample with petroleum jelly are shown. Some of these locations have petroleum jelly present while others do not, similar to the Semtex example shown earlier. The dataset for this example is a matrix with the rows corresponding to each spectra and the columns to each wavenumber and the entries to detected power. On applying PCA to this dataset each PC will be generated according to Equation 2.1. Plotting



Figure 2.3. Example of PCA applied to a 2-D dataset with x = y. In (a) the data is essentially 1-D as PC1 describes almost all the data. In (b) use of only PC1 results in a loss of information.

 w_1 (the first principal components weights) versus wavenumber generates the plot in Figure 2.4b and similarly w_2 versus wavenumber gives 2.4c. What can be seen then is that these plots of weightings versus wavenumbers are in fact spectra themselves and due to the nature of PCA correspond to the spectral features that change most throughout a dataset. These spectra can then be identified and attributed to certain substances. However, what can be seen in this example is that PCA will also capture the variance arising from differing powers. This is mostly apparent in 2.4b where the spectra looks very similar to the power baseline seen throughout the spectra in 2.4a. This does not contain useful spectral information and should be eliminated. The next section explains how to eliminate the influence of power variation alongside other interferents.

2.2.2 Derivative spectroscopy and Savitzky-Golay filtering

Often is the case in analyzing spectra that the features of interest are convoluted with that of interferents. In the case of interest RDX tends to have narrow spectral peaks, $\approx 20 \ cm^{-1}$, compared to the broad backgrounds of the substrate, e.g. plastic.



Figure 2.4. PCA example applied to a petroleum jelly sample. (a) shows multiple spectra measured from differing physical locations on the sample. PCA analysis is performed on this dataset and the first PC and second PC are shown in (b) and (c), respectively.

Derivative spectroscopy allows one to amplify the quickly varying features relative to the slowly varying background [47, 13], thus separating the substance spectra from both interferents and background power variations. While derivative spectroscopy is simple in concept, difficulties arise because real signals are not devoid of noise and this noise will completely dominate the derivative spectra as shown in the top curves of Figures 2.5b and 2.5c. In this example a signal was simulated with Lorentzian peaks on a broad Gaussian background. We wish to isolate the peaks from the background by using derivative spectroscopy. However, as can be seen when doing so without any smoothing the derivative signals are completely incomprehensible. Thus, in order for derivative spectra to be used the signal must be smoothed. While multiple filter techniques could be used, a particular filter known as the Savitzky-Golay (SG) filter is well suited as it accomplishes the smoothing and derivative processes simultaneously. The SG filtering technique is described next.

A common method used in commercial FTIR systems for smoothing spectra is the use of the SG filter. This filter was discovered by Savitzky and Golay in 1964 and they showed that their filter is equivalent to a moving least-squares error polynomial fit of order O and window length L [52]. This technique is visualized in Figure ??



Figure 2.5. Example of SG filtering. (a) Original Signal, (b) First derivative,
(c) Second derivative. In each plot the top signal is that of the simulated signal shifted by an arbitrary amount to aid in viewing. The bottom signal, marked with x's, is that of the SG filtered signal. All signals were normalized. a.u. is arbitrary units.

where each data point is fitted to a local polynomial of order O = 2 influenced by data points ranging within the window length L = 5 [54]. In showing this mathematically we will consider without lose of generality each fit window to be centered at n = 0. The fit is conducted over the domain $n \in [(L-1)/2, (L-1)/2]$. The polynomial fitted range \bar{x}_n will have the form,

$$\bar{x}_n = a_0 + a_1 n + a_2 n^2 + \dots + a_O n^O \tag{2.3}$$

With a_f corresponding to the f^{th} fitting coefficient found by least-squares error minimization. Evaluating 2.3 at n = 0, one finds the center point to have fitted value of a_0 . The window is then shifted to the right by one data point and fitted again. This is then repeated for the entirety of the signal.

The implementation of this smoothing technique can be done much more efficiently then suggested above due to its equivalence to SG filtering. It can be shown that finding the polynomial coefficients is equivalent to solving the matrix multiplication



Figure 2.6. Illustration of least-squares smoothing by locally fitting a second-degree polynomial (solid line) to five input samples.[54].

problem of

$$\mathbf{a} = (\mathbf{A}^{\mathbf{T}} \mathbf{A}^{-1}) \mathbf{A}^{\mathbf{T}} \mathbf{x}$$
(2.4)

where **a** is the coefficient vector, x is the data vector spanning the fitting window, and **A** is called the design matrix with elements α such that [55, 38]

$$\alpha_{n,i} = n^i, \quad -(L-1)/2 \le n \le (L-1)/2,$$

 $i = 0, 1, 2, 3, ..., O$
(2.5)

The central element of a is then the fitted value and is equivalent to evaluating \bar{x}_n in equation 2.3 at n = 0. A convenient aspect of the SG filter is that when filtering the signal one additionally obtains the values of the smoothed derivatives up to the order of one's fitting polynomial. Taking the j^{th} derivative, with $j \leq O$, and evaluating at n = 0 one obtains,

$$\left. \frac{\partial^j \bar{x}_n}{\partial n^j} \right|_{n=0} = j! a_j \tag{2.6}$$

This shows that solving equation 2.4 gives not only the fitted values of the original data, but also the derivatives of the fitted data as well. The bottom curves of Fig.

2.5 show the benefits of SG filtering on (a) raw spectra, (b) first derivative, and (c) second derivative. The question of what the optimum filter order and window length is not one that is easily answered and is the topic of much literature.[35, 33]. For the results shown here the order and length were selected by trial and error.

2.2.3 Results of chemometric analysis applied to explosive spectra

PCA following SG-second derivative filtering was applied to the RDX comprised explosives Semtex and C4. The second PC of the second-derivative spectra of C4 and Semtex are shown in 2.7. It can be seen that both spectra exhibit the same peak at \approx 1260 cm⁻¹ while only Semtex exhibits the peak doublet at 1096, and 1016 cm⁻¹. This shared peak is expected as both of these plastic explosives are derived from the same base explosive, RDX. While the RDX peak is in approximately the same location in both spectra, this peak is shifted upwards by 11 cm⁻¹ in the Semtex spectrum relative to C4.



Figure 2.7. Second derivative first principal component of C4 and Semtex. (b) displays a zoomed in version of (a) to show the 11 cm^{-1} shift.

An experiment was conducted to test if PCA could properly discriminate Semtex residues from bare plastic. The results are shown in Figure 2.8. The PC scores are plotted for varying samples/positions. Sample numbers 1-5 in the figure were taken from a background sample of plastic containing no explosive residue. Sample numbers 6-15 were obtained by scanning across a visually apparent spot of Semtex on the same plastic. The combination of these spectral processing techniques were capable of properly segmenting the individual measurements, with a positive value being no explosives and a negative value corresponding to the presence of Semtex. It can also be seen that there is a gradient present across the explosive spot with the largest negative value being in the center. This is also presumably the location of highest concentration and this result verifies that.



Figure 2.8. Second derivatives' first PC coordinates for a test of determining Semtex from a plastic substrate. Values 1-5 were obtained on a background with no explosive present. From 6-15 the illumination spot was scanned from just outside, across, and again outside a visible area of Semtex on plastic.

2.3 GUI tool development and application of chemometric techniques to array imaging

Application of PCA to discriminate thermal changes seen by the SEEK Thermal camera was tested by imaging a piece of cardboard illuminated by a QCL. Images were taken with the QCL and ran at multiple currents, thus changing the wavelength and power. The results are shown below in a tool developed to rapidly investigate the PC space, seen in Figure 2.9. The x and y axes are the first and second PC scores, respectively. The circle selects data having those PC scores and highlights them in the corresponding image. The first PC (PC1) explains 94 % of the variance in the data and, thus, explains almost all the change. PC1 has been attributed to the power change of the QCL, as it very cleanly discriminates the spot from the rest of the scene. The first figure selects the points on the negative side of PC1 and correspondingly highlights all the points outside of the spot. The second figure does the exact opposite, selecting those on the positive side of the axis and highlighting the QCL spot.

In addition to the tool developed for PCA imaging, a tool was developed to streamline the data analysis process outlined throughout this chapter. This tool is shown in Figure 2.10

2.4 Conclusion

The process of second-derivate SG filtering a MIR spectra and analyzing it using PCA has been able to successfully identify RDX on a plastic background. From theses results, 1260 cm^{-1} has been identified to be the wavenumber of particular interest for detection of RDX comprised explosives. Using a QCL centered at 1260 cm^{-1} a detection system can now be designed. The system can be of two types, a laser array type or a scanning type. A laser array type would require a minimum of two lasers, one with a wavenumber at the peak of interest and one located off this peak. Taking


Figure 2.9. MATLAB GUI tool for the application of PCA to array imaging. Pixels within the circle are highlighted in yellow. It is clear from the images that we are able to section out the beam (b) from the background (a). The dark blue pixels that are not selected in either image are the grid pixels in the compact that do not provide useful readings.



Figure 2.10. MATLAB data Analysis tool for the rapid application of the chemometric techniques outlined in this chapter

a ratiometric measurement at each point on the surface under interrogation would allow for explosive identification. A scanning system would sweep across a range of wavenumbers generating spectra similar to the FTIR spectra presented here.

The question of array system versus scanning system is primarily one of cost. An array system's base cost is much lower than a scanning system, but grows with the addition of QCLs. The need for multiple QCLs can be seen by inspecting the zoomed-in spectra shown in Figure 2.7. It is shown in this figure that even explosive compounds containing the same HEM can exhibit variation in spectral features. This shift of 11 cm^{-1} is a large shift when compared to the linewidth of a QCL. However, QCLs exhibit some spectral tuning range with temperature and current and this could be utilized to lessen the number of QCLs required. Because this project looks to reduce the cost of an explosive detection system as much as possible, an array system with the fewest number of QCLs is desired for the final system.

CHAPTER 3

CAMERA CHARACTERIZATION AND PRELIMINARY IMAGING RESULTS

In this chapter we evaluate the SEEK Compact as a representative VOx microbolometer for use as a scientific instrument. In order to be able to compare this device with other detectors, common sensitivity and noise metrics must be used. The metrics used are the noise-equivalent-differential temperature (NEDT), responsivity spectra and Allan variance. These metrics characterize the noise, responsivity, and performance stability over time, respectively. Each metric will be explained in further detail in their following sections. For all the following measurement experiments, a SEEK Compact device was used with a 12 μ m pixel pitch arranged in a 156 × 206 array with 93% of the pixels providing temperature readings. The device was connected to a PC via universal serial bus connection; device communication and data extraction was performed via Python script [58]. Image acquisition rate was measured to be 7 Hz.

3.1 Camera responsivity spectrum

A camera's responsivity spectra quantifies the amount of optical power incident on the camera that is converted to useful signal. As with the spectra shown in Chapter 2, one expects the results to be a strong function of wavelength. This comes primarily from the absorption efficiency of the camera material, in this case VOx. The SEEK Compact's responsivity spectrum was measured by directly imaging the output interferometer beam of a Bruker Vertex 80V FTIR spectrometer (silicon carbide globar source), thus recording an interferogram at each pixel, Figure 3.1.



Figure 3.1. Raw interferogram collected from SEEK Comapact FTIR experiment. The FFT is taken of this data to produce the resulting spectrum.

The FTIR was operated in step scan mode with 16 $\rm cm^{-1}$ resolution. Fifty images were taken at each mirror step and averaged together to create the interferograms for each of the $\approx 30,000$ pixels. The FFT of each interferogram was taken with a zero padding factor of two and the spectra normalized to its value at 1049 cm^{-1} . The resulting responsivity spectrum is presented in Figure 3.2 with the black trace representing the mean spectrum averaged over all pixels; the gray area represents \pm one standard deviation about the mean. The full width at half max (FWHM) of the spectra ranges from $\sigma = 833 - 1358 \text{ cm}^{-1}$. Figure 3.2 shows generally little variance between pixels. Within the usable region (i.e., FWHM) of the spectra, the standard deviation between pixels is on average 2.8% of the mean and never greater than 5.4% of the mean. The region spanned by the responsivity spectra is of great importance for molecular imaging and sensing applications as it lies in both the molecular "fingerprint" region and the second atmospheric transmission window [8, 37]. Molecules that one is capable of seeing in this region include: C-C stretch protein backbone ($\sigma = 930 - 939 cm^{-1}$), phospholipid ($\sigma = 978 cm^{-1}$), DNA/RNA $(\sigma = 1090 \text{ cm}^{-1})$, aromatic amino acids $(\sigma = 1162 - 1211 \text{ cm}^{-1})$, and nucleic acids $(\sigma = 1291 - 1324 \text{ cm}^{-1})$ [8]. Many energetic compounds also exhibit resonances in this range, such as PETN ($\sigma = 1279 \text{ cm}^{-1}$) and RDX ($\sigma = 1272 \text{ cm}^{-1}$) [25]. This

suggests that further research can be done with inexpensive VOx microbolometers being integrated as the detector in molecular and/or stand-off detection platforms.



Figure 3.2. Responsivity spectra of SEEK Compact. Black line: mean response; gray shade represents \pm one population standard deviation.

3.1.1 Noise equivalent differential temperature (NEDT)

Bolometer arrays are commonly used in thermography to estimate the temperature of an approximate black-body thermal source from detected LWIR power. The noise-limited minimum detectable temperature difference therefore defines a thermal detector's sensitivity, and is defined mathematically as:

$$NEDT = \frac{\Delta T}{P_s/P_n} \tag{3.1}$$

where ΔT is the temperature of the thermal source above some reference (e.g., room temperature), P_s is the differential (e.g., above room temperature) pixel signal, and P_n is the pixel's root-mean-square (RMS) noise value. To measure NEDT, a strip of high-emissivity 3M Scotch Super 88 Vinyl electrical tape [3] was placed on a copper heat block. The heat block's temperature was controlled via a microcontroller, and the temperature verified by a thermistor. The electrical tape was imaged at 14 different temperatures. Images of P_s at the 14 temperatures were found by subtracting a room temperature image from each image. P_n was measured by placing the SEEK Compact inside of a high emissivity cavity at room temperature and taking sequential images for ≈ 1.5 hours to find the signal standard deviation of each pixel. In order to remove any contributions to P_n from baseline drift over 1.5 hours, the signal of each pixel was fit to a 2^{nd} order polynomial which was subtracted from the raw data. The drift-subtracted P_n is used as the noise figure for all calculations, thereby approximating noise as temperature independent. From these experiments, the NEDT of each pixel can be calculated, as demonstrated in Fig. 3.3 for an object at $T = 30^{\circ}$ C when integrated for 3.57 s. NEDT histograms generated from images integrated at three integration times is presented in Fig. 3.4.



Figure 3.3. NEDT after 3.57 second integration time of SEEK Compact.



Figure 3.4. Pixel NEDT distribution versus integration time

Due to the stochastic nature of the averaging, the distribution mean and waist decreases with integration time. At the maximum SEEK Compact frame rate (7 Hz), the mean NEDT was 148 mK. The mean NEDT versus integration time is calculated from the histograms and presented on a log-log scale in Fig. 3.5. The dashed line on Fig. 3.5 represents a slope of -1/2, corresponding to NEDT decreasing with $1/\sqrt{T_I}$, where T_I is the integration time. The functional dependence of NEDT on $1/\sqrt{T_I}$ is characteristic of 1/f noise noise which has been reported as the dominant microbolometer noise source [31, 53]. Fig. 3.5 shows that the Compact is capable of reaching a NEDT at 30°C of 54 mK at 0.71 s and 25 mK at 3.43 s. These are comparable to the single frame NEDT of larger pitch microbolometer imaging systems and MCT (140K cooled) detectors respectively [49]. The FLIR T440 (FLIR[®] Systems, Inc) is a microbolometer camera commercially available for \approx \$12,000. The T440 is capable of imaging a NEDT at 30°C of 45mK at a frame rate of 60 Hz. Assuming the T440 is 1/f noise limited, and setting the NEDTs of the Compact equal to that of the T440, allows one to find that the SEEK Compact will require 93 times longer than the T440 in order to reach the same NEDT.



Figure 3.5. Mean NEDT vs integration time of SEEK Compact. Experimental data (solid line) follows a slope of -1/2 (dashed line).

3.1.2 Allan variance

To investigate measurement stability over time, Allan variance (two-sample variance) analysis [1, 2] was conducted. The Allan variance of a signal is the expected variance of two contiguous samples averaged over the time τ [50] and is computed as:

$$\sigma_y^2(\tau) = \mathbb{E}\{[\bar{y}_{k+1} - \bar{y}_k]^2\}$$
(3.2)

where \bar{y}_n is the $n^{th} \tau$ averaged data point. The raw data is the same as used in the experiment for measuring P_n . Results for five representative pixels are presented in Fig. 3.6. The slope of log-Allan variance versus log-integration time is -2 indicating that 1/f noise dominates for the entire experiment [1], up to 10 minutes of continuous integration. This long-term optical response stability indicates no long-term drift is present when imaging for reasonable image integration times.

3.1.3 Hyperspectral FTIR imaging

A potentially powerful application of low-cost bolomoter arrays is active LWIR vibrational spectroscopic imaging. We demonstrated hyperspectral LWIR imaging using a SEEK Compact as the detector array in an FTIR spectroscopic imaging



Figure 3.6. Allan variance of SEEK Compact for 5 representative pixels.

experiment. The experiment is the same as used to measure detector spectral response, but with a target sample placed in the FTIR beam path. The target was silicon nitride deposited by PECVD in the shape of an "N" and a "D" on a germanium wafer, Fig 3.7 (inset). The silicon nitride height of the "N" and "D" was 1μ m and 2μ m, respectively. The calculated transmission spectra for each pixel was normalized to that of a pixel corresponding to the bare germanium substrate in order to remove source, camera, and germanium absorption spectra from the calculated spectrum.

Fig. 3.7 shows the average background subtracted absorption spectra extracted from the SEEK Compact interferogram images for a single pixel in the "N," solid blue line, and the "D," dashed red line. As expected, SiN transmission increases with wavenumber between 900 to 1100 cm⁻¹ [7]. The scaling factor of ≈ 2 between the two curves is attributed to the silicon nitride height of the "D" being twice that of the "N," and therefore exhibiting twice the absorption.

Fig. 3.8 shows differential transmission images of the sample at 1024 cm^{-1} (top) and 1154 cm^{-1} (bottom). The values are normalized for each pixel to the measured transmission signal at 1096 cm⁻¹ for that same pixel, and therefore representing a differential optical absorption spectroscopy (DOAS) measurement [44]. While the DOAS image at 1024 cm⁻¹ shows little difference between the "N" and the "D," a visible difference is present at 1154 cm⁻¹. Since spectral features due to the germanium



Figure 3.7. Single pixel spectra extracted from SEEK Thermal hyperspectral FTIR imaging data. Blue solid line: 1 μ m thick silicon nitride ("N"); Red dashed line: 2 μ m thick silicon nitride ("D").

substrate, camera responsivity, IR source spectrum, and silicon nitride absorption have been divided out, this difference can be attributed to other spectroscopically relevant factors such as a Fabry-perot effect or variation in material quality between the "N" and "D," which were deposited in two separate fabrication steps. In total, these results demonstrate proof-of-concept hyperspectral imaging using a commercially available and low-cost VOx microbolometer array.

3.2 Conclusion

This work characterized the SEEK Compact, a commercially available and low cost VOx microbolomter array, for use in LWIR hyperspectral imaging. The SEEK Compact has a single frame NEDT of 148 mK in 0.15 s. Comparing this to a research grade and commercially available FLIR T440 thermal imaging systems shows a decrease in price and weight by a factor of 48 (\$12000/\$250) and 4 (2.0 lbs/0.5 lbs), respectively, with an increase in image acquisition time by a factor of 93 for the same NEDT. Therefore, such detectors can enable applications where field deployable imaging is required and reduced sensitivity can be overcome via active illumination or complimentary imaging techniques.



Figure 3.8. Differential optical absorption spectroscopy images relative to 1096 cm⁻¹ at 1024 cm⁻¹ (top) and 1154 cm⁻¹ (bottom). "N" and "D" are 1 μ m and 2 μ m thick silicon nitride, respectively, deposited in germanium.

CHAPTER 4

CAMERA SOFTWARE MODIFICATIONS AND SYSTEM DEVELOPMENT

With the knowledge of the noise characteristics and region of spectral interest identified, an explosive detection system has begun to be realized in a laboratory setup to begin testing the SEEK compact as the detector in a trace explosive detection platform. This process of designing and implementing an explosives detection system with a VOx microbolometer is outlined in this chapter beginning with the high level system design showing the required hardware and layout. Then software modifications are presented that have allowed the SEEK Compact to greatly exceed the 7 Hz operation and reach a frame rate of 32 Hz. This greatly lowers the trade-off presented in Chapter 3 and makes using the SEEK Compact for scientific applications even more attractive. Finally, the data acquisition, GUI design, and client-server operation are presented.

4.1 High level system design

An explosives detection system has been realized by integrating a SEEK Compact VOx microbolometer camera with a remote WIFI hotspot provided by a Raspberry Pi (Rpi) and peripheral hardware. The hardware layout is shown in Figure 4.1. The illumination source is a MIRCAT tunable QCL from Daylight Solutions. This laser covers the spectral region from 1150 cm^{-1} to 1310 cm^{-1} reaching peak output power of 370 mW at 1260 cm^{-1} , coinciding with our desired wavelength of detection for RDX. The minimum power across this spectral range is 50 mW at the limits of the spectral range. In order to reach these powers the MIRCAT must be running in



Figure 4.1. Cartoon of the experimental setup realized for testing the SEEK Compact as the detector in a LWIR trace explosives setup.

continuous-wave mode and requires cooling. To maintain the temperature at 20 C a water-cooled Oasis UC 160 was chosen providing 160 W of cooling power. The MIRCat's beam spot size is roughly 1 mm in diameter and is not adequately large for a non-scanning system configuration. In order to image a larger portion of the sample a telescopic magnifying lens setup has been used with a magnification of ≈ 25 , bringing the beam spot size to roughly an inch in diameter. The sample is then placed in the path of the beam slightly off axis in order to minimize back reflections directly into the laser. The bolometer is placed within 45° of the sample's surface normal in order to collect some of the diffusely scattered light. Currently, the bolometer is within 6 in of the lens. If one wishes to lengthen this observation distance magnifying optics must also be placed on the observing end.

4.2 Software modifications

Building off of the initial code used in Chapter 2 and 3, it was discovered that the camera has different operation modes that can be selected. When normal phone operation is initiated the camera begins a repeating pattern of closing the shutter for a frame, measuring the background signal (i.e. bolometer dark current) on the next frame, open the shutter and wait one frame, and image eight background subtracted frames. This was the operation mode used in the characterization of the camera in Chapter 3 and has a frame rate of 7 Hz. In addition to background subtraction, studying the data showed that the camera is also doing additional post-processing that is some unknown combination of scaling and offsetting. This changes with the dynamic range of the scene and since an exact knowledge of this post-processing is unknown, it is detrimental to our understanding of the data. However, through studying the signals sent to the camera during initialization it was discovered that there exists more operating modes. While most of them are not useful one of them allows the camera to run at 32 Hz, but with the shutter closed. Unfortunately, a programmatic way of opening the shutter is still not known to the author so the device was taken apart to study. It was discovered that the shutter is magnetically operated by a nearby current loop and defaults closed. When the loop is energized the magnetic field flips the shutter open. To replicate this effect the addition of a simple magnet adhered to the back of the camera was enough to hold the shutter open permanently and allows for the 32 Hz framerate mode to be utilized. The one tradeoff that has arisen from not using the camera's built in post-processing mode is that the pixel values drift. This pixel value drift becomes less and less prominent as the camera is imaging longer and, therefore, is most likely due to the camera warming up. This means that to image immediately after the camera has been turned on backgrounds will have to be taken more frequently. This discovery greatly alters the trade-off reported earlier of 48x cost reduction for a 93x longer integration time to only a 20.43x rate.

4.3 Data acquisition, GUI design, and client-server operation

The SEEK Compact microbolometer in this system is controlled via a USB connection to a RPi. The code was designed such that this RPi acts as a server that waits for requests from a client, either wirelessly or via USB. Once the code has begun

running the RPi first initializes a connection to the client either via websockets if wireless or usb. After connection is established, commands are sent to the SEEK Compact to run the microbolometer through a brief initialization sequence to prepare for measurements. The RPi then begins alternating between sending measurement commands to the microbolometer, retrieving the raw images, and pushing the data to the client. On the client side the data must be unpacked from its original format as 16 bit little endian into the scene image and displayed. To display and control the image a TKinter GUI was created. To test the capabilities of the wireless configuration a prototype system was created utilizing a RF trasnmitter/receiver box developed by Professor Chisum and his research group. Since this box uses a RPi, bringing the box up to operating conditions is as simple as inserting an SD card with the code on it. Figure 4.2 outlines the client-server operation.



Figure 4.2. Schematic of client-server operation for the trace detection system. Requests are sent from the client to the server that control the bolometer. The bolometer then sends raw images to the server that are pushed to the client.

Initially the GUI utilized the standard matplotlib backend for data visualization, but it limited the framerate to roughly 5 Hz due to the slow graphic rendering. In order to improve the framerate the backend was changed to QT achieving a live viewing framerate of 16 Hz, still half that of the maximum imaging rate. The current speed bottleneck arises from the sequential implementation of the data unpacking and display processes. This framerate limitation could certainly be improved upon by threading theses two processes and running them in parallel, but this has not been implemented as of yet. However, if one is interested not in live imaging but only data capture the display can be turned off and 32 Hz imaging is once again available. This imaging mode is more likely the scenario that will be utilized in the field as it is generally desirable to integrate the data for a set amount of time to increase SNR and then display a single image. In addition to being faster than the matplotlib backend, the QT GUI also comes with data analysis tools such as region of interest (ROI) and histogram already built. The realized GUI is shown in Figure 4.3. On the right side of the image the histogram and range adjusting tool is shown. The spike at zero in the histogram corresponds to the 7% of pixels that do not provide useful temperature readings. These pixels are arranged in a uniform grid pattern and can be seen on the image as well. Additionally shown is the ROI selection and plotting tool that allows one to generate a plot of pixel values in the ROI (area in the rectangle). Finally, the GUI has basic saving features allow the user to designate the number of frames to be recorded and also allows the user to update the background at any time to compensate for pixel drift.

4.4 Conclusion

In this chapter an experimental setup for testing the SEEK Compact VOx microbolometer as the detector in an explosive detection platform was outlined. This setup utilizes a high-power tunable laser centered at the RDX wavenumber of 1260 cm^{-1} . Additionally, software modifications were made in order to increase the imaging framerate of the Compact from 7 Hz to 32 Hz. This modification alone greatly increases the feasibility of using the Compact as a detector as it will reduce the imaging time by a factor of 4.5. In terms of the trade-offs presented in Chapter 3 this puts the compact at 48x cost reduction 20.43x rate. The camera is controlled via a RPi acting as a server that waits for commands either wirelessly or via USB. A GUI was also created to make use of the camera extremely easy to use and has a live viewing rate of 16 Hz, but can also record data at a frame rate of 32 Hz.



Figure 4.3. GUI designed for live viewing of output of SEEK Compact. Currently, live feed can be viewed at 16 Hz. 32 Hz non live operation is also available. Shown on the display is the ROI and histogram tools.

CHAPTER 5

INITIAL MEASUREMENT RESULTS AND FUTURE WORK

In this chapter initial measurements from the system outlined in the previous chapter are presented. From these initial results it becomes apparent that the coherent imaging phenomena speckle is degrading the current quality of the images obtained. Ways of combating speckle are outlined. Additionally, a discussion of the on-going collaboration with Professor Vijay Gupta and student Bernardo Aquino in the development of optimal sensor selection algorithms is addressed.

5.1 Initial measurement results

To test the developed trace detection system a ratiometric imaging experiment was conducted with a sample of Crisco vegetable shortening on a glass slide. The wavenumbers used were 1234 cm^{-1} and 1270 cm^{-1} . These were chosen from measurements of the Crisco spectrum by an FTIR. Additionally, the power curves of the MIRcat were measured at the two wavelengths and the currents selected such that the powers were the same. The collected sample images are shown in Figure 5.1 with the top image corresponding to 1234 cm^{-1} and the bottom to 1270 cm^{-1} . What can be seen by comparing the two histograms is that there is a slight shift in the overall pixel distribution between the two. The ratio of these images is also shown in Figure 5.2. While one can see a light blue shape corresponding to the Crisco smear, it is covered in very bright pixels scattered throughout making it difficult to discern. It is believed that these bright spots arise from speckling. Speckling is a phenomena that occurs when imaging with a coherent imaging system, such as a laser. It arises



Figure 5.1. Image of glass slide smeared with Crisco at two different wavenumbers 1234 cm^{-1} and 1270 cm^{-1} . On the right is the histogram of the two images. The peaks at zero are non-useful pixels.



Figure 5.2. Ratio of the two images shown in Figure 5.1. While the Crisco smear is still visible as a light blue, it is covered in bright spots that have been attributed to speckle.

from the the non-uniformity of the sample's surface resulting in diffuse reflections being sent in every direction. These reflections will have undergone different pathlengths and will interfere causing randomly distributed bright and dark spots across the image. Speckle can be identified by the peculiar fact that the speckles come into focus as the scene itself is shifted out of focus, as shown in Figure 5.3. Other explosive detection groups have identified speckling as a significant problem in their systems with one group reporting that speckle accounted for 50% of their observed signal [26, 23, 39].



Figure 5.3. Example of speckle in coherent imaging. The left image shows that the speckle pattern becomes more distinguished as the image is out of focus.

In order to reduce speckle one must break the laser's coherence. There exist a handful of ways to lower the coherence of a laser, but the most simple is through a spinning disk diffuser. A diffuser is a rough piece of reflective or transmissive material that upon allowing EMR to reflect off of or transmit through generates a diffuse reflection pattern similar to that seen when using a laser pointer on a projector screen. If this EMR is then collected and collimated into a beam it is found that this beam exhibits lower coherence than the original beam. Images obtained with this beam will exhibit less speckling as compared to those obtained with the original highly coherent beam. One can reduce speckling even further by leveraging the finite integration time of one's camera. Since the diffuser's surface is essentially randomly configured, if the diffuser is rotated to a different orientation the generated speckle pattern seen at the camera is also altered randomly. By spinning the diffuser at a rate much faster than the frame rate of the camera, the images generated are an average of all these different speckle patterns. Since these speckle patterns are random they will average out to the true image.

Due to most substance being opaque in the LWIR, reflection configurations are generally used. Notable material exceptions being poly-crystalline diamond and zinc-selenide. For reflection setups gold coated mirrors are typically used as they have very high reflection in this spectral region. The difficulty in using reflection setups for imaging lies in the fact that collecting the diffusely scattered EMR is very difficult, which causes a drastic decrease in available power. To counter this power loss researchers have begun designing integrating cavities to place the diffusers inside of [22].

To alleviate speckling in the trace detection system a spinning disk diffuser reflection setup will be implemented. While the optical throughput of this system is low, for short distances and high input powers, as is the case for the laboratory system designed in Chapter 4, the power should be sufficient. However, it is acknowledged that this will not be sufficient for long distance stand-off detection. In the case that the gold-disk diffuser throughput is insufficient alternatives will have to be investigated. A possible alternative is that of zinc-selenide transmissive diffusers. These, however, are much more expensive than the gold-disk diffusers.

5.2 Optimal sensor selection algorithm collaboration

As briefly stated in the introduction, the ever-changing spectral data received from varying substrates, substances, and experimental conditions has made explosives detection difficult. In order to combat this many groups have switched their efforts to developing detection algorithms. Similar methods as presented in Ch.2 were attempted and found that while PCA was very good in laboratory settings where the samples are highly controlled, its selectivity was lacking in real-world situations. These results have prompted the development of more sophisticated algorithms including machine learning and Bayesian techniques. As more sophisticated algorithm development is out of the author's area of expertise, a collaboration with Professor Gupta and his student Berando Aquino was established. In Professor Gupta's previous work he and his graduate student developed a method for determining optimal sensor selection. Optimal sensor selection solves the problem of given a set of sensors that take individual (possibly correlated) measurements, how does one determine what sensors to use, in what order and frequency, to most efficiently come to a conclusion with a given confidence. Sensor is not to be taken in a literal sense, but simply as any unique measurement. For example, an image taken at a single illumination wavelength is a sensor. The proposed future work is to develop a multi-modal system capable of obtaining many different measurements to leverage the previous work by Professor Gupta. This would lead to a feed back loop where measurements are provided to test if they improve the performance of the detection algorithms, while the algorithms can act as a guide to the most efficient measurement scheme. Most importantly the algorithms can help answer what is the minimum number of discrete QCLs necessary for a laser array detection system.

This collaboration is on-going, where my role is to provide the data and conduct physical interpretations of the data to help guide Bernardo and Professor Gupta's analysis. Our chief insight is that a single image provides many independent experiments to classify a given object. Even if the correct classification probability of a single pixel is small due to low SNR, the presence of many different experiments can boost this probability to competitive standards. In other words, we move from the traditional one pixel, many wavenumbers approach to a many pixels, single wavenumber approach. This proposed method has a throughput higher than the traditional laser scan using multiple wavenumbers, since only one wavenumber is required for the classification. Moreover, it provides effective detection with low SWAP-C. While their is a limitation to how effectively one can discriminate via one wavelength we are working towards getting all available information out of as little experimental data as possible. This approach reduces the number of lasers required for real life systems lowering the cost further.

CHAPTER 6

CONCLUSION TO PART I

The first part of this report has presented work towards the development of a LWIR explosive detection platform utilizing a low-cost VOx microbolometer as the detector.

First, the spectra of the RDX comprised explosives C4 and Semtex were taken with an FTIR and an absorption peak at 1260 cm^{-1} was found to be a common feature. Using this peak the chemometric techniques of PCA and second-derivative SG filtering was shown to be able to be used to differentiate explosives from substrates.

Second, the SEEK Compact microbolometer was studied as a representative low-cost VOx detector. The performance characteristics measured were the responsivity spectrum, NEDT, and Allan variance. In addition, a thin-film hyperspectral imaging experiment was conducted to demonstrate the viability of using low-cost microbolometers for spectral imaging.

In chapter 4 camera software modifications and system design was outlined. A schematic of a laboratory testing system consisting of the MIRCat laser system from Daylight Solutions was presented. Most importantly this section showed that the camera's imaging rate could be increased from 7 Hz to 32 Hz greatly reducing the time required to reach a given NEDT. Also outlined was the data acquisition protocol, GUI design, and client server operation characteristics.

Initial measurements were presented to demonstrate the current imaging capabilities of this system and showed the current issue with speckle imaging and outlined potential solutions. The combination of speckle reduction and optimal sensor selection algorithms with a VOx microbolometer should allow for the realization of a low SWAP, low-cost trace explosives detection system for both checkpoint and stand-off applications. PART II:

TIME-HARMONIC (PHASOR) THERMAL IMAGING

CHAPTER 7

TIME-HARMONIC (PHASOR) THERMAL IMAGING

7.1 Introduction and motivation

The "flash method" is the standard way of measuring thermal properties of materials such as thermal diffusivity and thermal conductivity. This method belongs to the category of laser-based, non-destructive evaluation techniques [15, 14]. It is performed by using a laser to excite the material of one's interest from the front and a detector is placed on the reverse side of the material in order to measure the transient heat flow to the other side. While being very sensitive, these systems require large, complicated expensive machines usually utilizing LN_2 cooled IR detectors. In addition the flash method does not provide a straight-forward generalization to thermal imaging due to the fact that in the majority of imaging scenarios one does not have access to the back side of the object one wishes to image. In order to expand upon this method, this section investigates a phasor (time-harmonic) based laser excitation method in order to image and classify thermal properties of systems from a forward view.

This section begins with a first-principles derivation of the phasor solution for the standard heat equation. Next, an implicit, unconditionally stable numerical scheme is outlined to simulate the phasor response of materials with different diffusivitives (D) and thermal conductivities (k), including simulating heterogenous samples. Laboratory experimental results follow verifying the simulation results. Finally, this section concludes with a brief discussion of extending the work shown here into full-fledged diffuse thermal tomography.

7.2 Mathematical description of problem

7.2.1 Casting of phasor problem

The system of interest will be that of the standard homogenous heat equation, initial-boundary valued problem. This is presented below.

$$\frac{\partial u(x,t)}{\partial t} = D \frac{\partial^2 u(x,t)}{\partial x^2}$$
(7.1)

$$-k\frac{\partial u(0,t)}{\partial x} + hu(0,t) = \phi_{left}(t)$$
(7.2)

$$k\frac{\partial u(L,t)}{\partial x} + hu(L,t) = \phi_{right}(t)$$
(7.3)

$$u(x,0) = \psi(x) \tag{7.4}$$

$$0 \le x \le L, \quad t \ge 0, \quad u(x,t) \in \mathbb{R}$$
(7.5)

where u(x,t) is the temperature at position x and time t, D is the diffusivity, k is the conductivity, h is the coefficient of convection, $\phi_{left}(t)$ and $\phi_{right}(t)$ are heat source terms, $\phi(x)$ is the initial condition and, L is the length of the homogenous sample. The boundary conditions, therefore, take into account both conduction away from the surface towards the interior of the material, and convection into the surrounding environment whose temperature is by definition 0. It will be assumed that $\phi_{left} = \Re(e^{i\omega t})$ and, therefore, as $t \to \infty$, $u(x,t) \to \Re(U(x)e^{i\omega t})$, regardless of initial condition $\psi(x)$. Additionally, that $\phi_{right} = 0$.

Therefore, the above initial-boundary value problem becomes,

$$i\omega U(x) = D \frac{\partial^2 U(x)}{\partial x^2}$$
(7.6)

$$-k\frac{\partial U(0)}{\partial x} + hU(0) = 1 \tag{7.7}$$

$$k\frac{\partial U(L)}{\partial x} + hU(L) = 0 \tag{7.8}$$

$$0 \le x \le L, \quad t \ge 0, \quad U(x) \in \mathbb{C}$$
(7.9)

Using the identity $2i = (1+i)^2$, 7.6 can be rewritten as,

$$\frac{\partial^2 U(x)}{\partial x^2} - ((1+i)\sqrt{\frac{\omega}{2D}})^2 U(x) = 0.$$
 (7.10)

Eq. 7.10 has two cases of solutions: $\omega = 0$ and $\omega \neq 0$. If $\omega = 0, 7.10$ becomes

$$\frac{\partial^2 U(x)}{\partial x^2} = 0, \tag{7.11}$$

with corresponding solution,

$$U(x) = mx + b. \tag{7.12}$$

Since 7.12 must satisfy the boundary conditions of 7.7 and 7.8, one is led to the simultaneous equations,

$$\begin{cases} -km + hb = 1 \\ (k + hL)m + hb = 0. \end{cases}$$
(7.13)

This leads to,

$$m = \frac{-1}{hL - 2k}$$
, $b = \frac{k + hL}{2kh + h^2L}$. (7.14)

If $\omega \neq 0$, 7.10 has general solution,

$$U(x) = c_1 exp((1+i)\sqrt{\frac{\omega}{2D}}x) + c_2 exp(-(1+i)\sqrt{\frac{\omega}{2D}}x).$$
 (7.15)

As before, subjecting 7.10 to the boundary conditions of 7.7 and 7.8, one is led the simultaneous equations,

$$\begin{cases} c_1(-kp+h) + c_2(kp+h) = 1\\ c_1(kp+h)e^{pL} + c_2(-kp+h)e^{-pL} = 0 \end{cases}$$
(7.16)

where $p = (1+i)\sqrt{\frac{\omega}{2D}}$ has been substituted to simplify the notation. Finally, c_1 and c_2 are found to be,

$$c_1 = \frac{-kp+h}{(-kp+h)^2 - (kp+h)^2 e^{2pL}}$$
(7.17)

$$c_2 = \frac{-kp - h}{(-kp + h)^2 e^{-2pL} - (kp + h)^2}.$$
(7.18)

Thus concludes the two cases.

Since the system of interest is linear one can construct the solution to any arbitrary input function $\sum A(\omega)e^{i\omega t}$ by a standard Fourier construction.

7.2.2 Special case: |kp| >> |h|

It is assumed in this section, and for the rest of the paper, that |kp|>>|h| . Therefore, eqs. 7.17 and 7.18 become

$$c_1 = \frac{-1}{kp(1 - e^{2pL})} \tag{7.19}$$

$$c_2 = \frac{1}{kp(1 - e^{-2pL})}. (7.20)$$

Of interest is the magnitude and phase of c_1 and c_2 . Let us define the "characteristic diffusion time" as $t_c = L^2/D$ and the corresponding "characteristic diffusion radial frequency" as $w_c = 2\pi/t_c$. Then, 7.21 and 7.22 can be rewritten as,

$$c_1 = \frac{2Le^{i(3\pi/4 - tan^{-1}\beta_1)}}{ky\sqrt{1 - 2e^y \cos y + e^{2y}}}$$
(7.21)

$$c_2 = \frac{2Le^{i(\pi/4 - \tan^{-1}\beta_2)}}{ky\sqrt{1 - 2e^{-y}cosy + e^{-2y}}}$$
(7.22)

$$\beta_1 = \tan^{-1}(\frac{-\sin(y)}{e^y - \cos(y)}) \tag{7.23}$$

$$\beta_2 = \tan^{-1}(\frac{\sin(y)}{e^y - \cos(y)}) \tag{7.24}$$

where $y = \sqrt{2\omega t_c} = 2\sqrt{\pi\omega/\omega_c}$.

It is instructive, at this point, to consider the value of the exponential functions versus length, L, and diffusivity, D. These have respective (approximate) thermal properties $D_{glass} = 3.4 \times 10^{-7} \ m^2/s$, $k_{glass} = 1.05 \ W/(m \cdot K)$ and $D_{copper} = 1 \times 10^{-4} \ m^2/s$, $k_{copper} = 401 \ W/(m \cdot K)$. What can be deduced from these values is that in every case apart from low frequency, thin samples of copper, $e^y >> 1$ and $e^{-y} << 1$. Therefore, if one operates at a high enough relative frequency, $c_1 \to 0$, $\beta_2 \to 0$, and c_2 becomes,

$$c_{2} = \frac{1}{k\sqrt{\frac{\omega}{2D}}(1+i)} = \frac{e^{-i\pi/4}}{k}\sqrt{\frac{D}{\omega}}$$
(7.25)

7.3 Numerical analysis

7.3.1 Numerical scheme

To verify the theory in the previous section a numerical-simulation is conducted. The numerical scheme used is that of the common Crank-Nicholson method used for the heat equation. This is an implicit numerical method in time and is, therefore, unconditionally stable. What, however, is not straight-forward is how to handle the boundary conditions and for that reason the numerical scheme is included here.

The first step is to discretize the non-homogenous (general) heat equation in time following the trapezoidal rule,

$$\frac{du(x,t)}{dt} = f(t,u) = \frac{\partial D(x)}{\partial x} \frac{\partial u(x,t)}{\partial x} + D(x) \frac{\partial^2 u(x,t)}{\partial x^2}$$
(7.26)

$$u^{n+1}(x) = u^n(x) + \frac{\Delta t}{2} (f(t^{n+1}, u^{n+1}) + f(t^n, u^n))$$
(7.27)

where time, t has been discretized into $\{t^n\} \in t, n \in \{0, 1, 2, ..., N - 1\}$ and $u(x, t^n)u^n(x)$. Next, the spatial derivatives in Eq. 7.26 are approximated by their appropriate central differences. This numerical scheme is, therefore, 1^{st} order accurate in both space and time. Discretizing the domain x into $\{x_i\} \in x, i \in \{0, 1, 2, ..., M - 1\}$ and defining $u(x_i)u_i$,

$$A_{i}u_{i+1}^{n+1} + B_{i}u_{i}^{n+1} + C_{i}u_{i-1}^{n+1} = \alpha_{i}u_{i+1}^{n} + \beta_{i}u_{i}^{n} + \gamma_{i}u_{i-1}^{n}.$$
(7.28)

$$A_i = -\frac{\Delta t}{4\Delta x} \frac{\partial D_i}{\partial x} - \frac{\Delta t}{2\Delta x^2} D_i$$
(7.29)

$$B_i = 1 + \frac{\Delta t}{\Delta x^2} D_i \tag{7.30}$$

$$C_i = \frac{\Delta t}{4\Delta x} \frac{\partial D_i}{\partial x} - \frac{\Delta t}{2\Delta x^2} D_i \tag{7.31}$$

$$\alpha_i = \frac{\Delta t}{4\Delta x} \frac{\partial D_i}{\partial x} - \frac{\Delta t}{2\Delta x^2} D_i \tag{7.32}$$

$$\beta_i = 1 - \frac{\Delta t}{\Delta x^2} D_i \tag{7.33}$$

$$\gamma_i = -\frac{\Delta t}{4\Delta x} \frac{\partial D_i}{\partial x} + \frac{\Delta t}{2\Delta x^2} D_i \tag{7.34}$$

This scheme works for all interior points $i \in \{1, 2, ..., M-2\}$, however, if one considers

the equations concerning the boundary points,

$$A_{0}u_{1}^{n+1} + B_{0}u_{0}^{n+1} + C_{0}u_{-1}^{n+1} = \alpha_{0}u_{1}^{n} + \beta_{0}u_{0}^{n} + \gamma_{0}u_{-1}^{n}$$

$$A_{M-1}u_{M}^{n+1} + B_{M-1}u_{M-1}^{n+1} + C_{M-1}u_{M-2}^{n+1} = \alpha_{M-1}u_{M}^{n} + \beta_{M-1}u_{M-1}^{n} + \gamma_{M-1}u_{M-2}^{n}.$$
(7.35)

one finds the need for the undefined points
$$u_M$$
 and u_{-1} . In an effort to define these
two points the boundary conditions are investigated using the same numerical scheme
as outlined above.

$$-k(0)\frac{\partial u(0,t)}{\partial x} + hu(0,t) = \phi_{left}(t)$$
(7.37)

$$-k_1 \frac{u_1^n - u_{-1}^n}{2\Delta x} + hu_0^n = \phi_{left}^n \tag{7.38}$$

(7.36)

$$u_{-1}^{n} = u_{1}^{n} + \frac{2\Delta x}{k_{0}}(\phi_{left} - hu_{0}^{n}).$$
(7.39)

Therefore, one can define an artificial point, known as a "ghost point" in the literature, u_{-1}^n such that it obeys the boundary conditions. Similarly for u_M ,

$$k(L)\frac{\partial u(L,t)}{\partial x} + hu(L,t) = \phi_{right}(t)$$
(7.40)

$$k_{M-1}\frac{u_M^n - u_{M-2}^n}{2\Delta x} + hu_{M-1}^n = \phi_{right}^n \tag{7.41}$$

$$u_M^n = u_{M-2}^n + \frac{2\Delta x}{k_{M-1}} (\phi_{right} - hu_0^n).$$
(7.42)

Let us define the MxM matrix T_1 as,

$$T_{1} \begin{bmatrix} B_{0} & A_{0} & 0 & 0 & \dots & 0 & 0 & 0 & 0 \\ C_{1} & B_{1} & A_{1} & 0 & \dots & 0 & 0 & 0 & 0 \\ 0 & C_{2} & B_{2} & A_{2} & \dots & 0 & 0 & 0 & 0 \\ \vdots & \vdots & \vdots & \vdots & \ddots & \vdots & \vdots & \vdots & \vdots \\ 0 & 0 & 0 & 0 & \dots & C_{M-3} & B_{M-3} & A_{M-3} & 0 \\ 0 & 0 & 0 & 0 & \dots & 0 & C_{M-2} & B_{M-2} & A_{M-2} \\ 0 & 0 & 0 & 0 & \dots & 0 & 0 & C_{M-1} & B_{M-1} \end{bmatrix}$$
(7.43)

and the matrix T_2 as the same form as T_1 exchanging $A_i \implies \alpha_i, B_i \implies \beta_i$, and $C_i \implies \gamma_i$. With these definitions the above set of equations can be written as,

$$T1\begin{bmatrix}u_{0}\\u_{1}\\\vdots\\u_{M-2}\\u_{M-1}\end{bmatrix}^{n+1} + \begin{bmatrix}C_{0}u_{-1}\\0\\\vdots\\0\\A_{M-1}u_{M}\end{bmatrix}^{n+1} = T2\begin{bmatrix}u_{0}\\u_{1}\\\vdots\\u_{M-2}\\u_{M-2}\\u_{M-1}\end{bmatrix}^{n} + \begin{bmatrix}\gamma_{0}u_{-1}\\0\\\vdots\\0\\\alpha_{M-1}u_{M}\end{bmatrix}^{n}$$
(7.44)

where the vectors $[C_0 u_{n-1}, 0, \dots, 0, A_{M-1} u_M]^T$ and $[\gamma_0 u_{n-1}, 0, \dots, 0, \gamma_{M-1} u_M]^T$ account for the boundary conditions. These boundary vectors can be rewritten to be a function of $\vec{u} = [u_0, u_1, \dots, u_{M-2}, u_{M-1}]^T$.

$$=\lambda_1^n \vec{u}^n + \Phi_1^n \tag{7.46}$$

Similarly we can define λ_2 and $\vec{\Phi}_2$ for the right-hand side of Eq. 7.44 resulting in the compact equation,

$$T_1\vec{u}^{n+1} + \lambda_1\vec{u}^{n+1} + \Phi_1^{n+1} = T_2\vec{u}^n + \lambda_2\vec{u}^n + \Phi_2^n$$
(7.47)

$$(T_1 + \lambda_1)\vec{u}^{n+1} + \Phi_1^{n+1} = (T_2 + \lambda_2)\vec{u}^n + \Phi_2^n$$
(7.48)

$$\vec{u}^{n+1} = (T_1 + \lambda_1)^{-1} (T_2 + \lambda_2) \vec{u}^n + (T_1 + \lambda_1)^{-1} (\Phi_2^n - \Phi_1^{n+1})$$
(7.49)

This is the final equation and outlines the algorithm for which given an initial-value, boundary problem, one can compute \vec{u}^{n+1} from \vec{u}^n . Particularly, it is brought to the reader's attention that since the user specifies the excitation, ϕ_{left} and ϕ_{right} (and, correspondingly, Φ_2 and Φ_1) are known a priori for all time. This allows for the existence of the Φ_1^{n+1} term on the right hand side of the equation.

Numerical Results 7.3.1.1

A numerical simulation is conducted following the above numerical scheme for two materials, copper and glass. In both cases the thickness of the material is taken to be 1 mm, $A = 1 W/m^2$, the excitation frequency is taken to be 7 $Hz \approx 44 rad/s$, and h
(coefficient of convection) is taken to be $100 W/(m^2 \cdot K)$ at either end. The results are presented in Fig. 7.1. Two types of plots are displayed; one is the surface temperature as a function of time u(0,t) (b and d) and the other is a time snap-shot of the spatial distribution at 1 second, u(x, 1 s) (a and c). One should be careful to notice that the scales are different on each image. This has been done in order to properly convey the spatial profile of copper which is indiscernible when plotted on the same scale as glass. There exists two curves on each plot. The first being the numerical result is the solid curve and the +-signs designating the approximate analytical result from eqs. 7.21 and 7.22. The agreement between the two is excellent, apart from the beginning of plot (b). This is due to the fact that the numerical result is starting from an initial condition of u(x, 0) = 0 while the analytical result explicitly assumes steady-state behavior. In fact it is evident that the numerical solution converges to the analytical solution.

Since the numerical method developed above started from the general heat equation and not the homogenous equation (which the analytical solution stemmed from) it can be directly applicable to any arbitrary diffusivity profile. Of interest will be the next simplest case after a homogenous sample, that of a two material stack. The system that will be discussed is schematically shown in fig. 7.2. This model does not contain any contribution from contact resistance.

Fig. 7.3 shows the numerical result for the simulation of composite materials made from copper and glass. In (a) one can notice that the surface profile of the simulate (blue) obtains a higher value at the surface than homogenous glass (red). In addition, there a discontinuity in temperature corresponding to the unit step. This is a well known phenomena in composite flux constant heating scenarios, but has not been reported in the time harmonic case to the author's knowledge. The size of this gap changes harmonically in time as well. In (b) there is an apparent increase in temperature, but this is actually due to the fact that the composite case is out



Figure 7.1. Numerical simulation results for time-harmonic excitation of homogenous samples of glass and copper. Shown are the spatial distributions in space at t = 1 second after turning on the excitation ((a) glass, (c) copper) and the the temporal profile at the surface x = 0 ((b) glass, (d) copper). The initial condition was taken to be u(x, 0) = 0 and that is why some discrepancy is noticed in (b) at the beginning, but disappears in steady-state. Copper's thermal properties make it so steady-state is almost instantaneously obtained.



Figure 7.2. Visualization of two-material case for numerical analysis

of phase with respect to the homogenous case (see figure (d)). There also exists a temperature build-up at x = 0 due to the insulating glass. The profile in the glass part resembles that the homogenous glass case as well (green). In plots (c) and (d) the surface temperature profile is shown. What can be seen in (c) is that the oscillations of the glass-copper case are massive compared to the original homogenous cases. This will be shown to be true in the experiments to follow and not simply a numerical effect. In (d) one sees that the copper-glass case is actually worse than either homogenous case. This shows that this systems are not invertible in space.

While this numerical example shows some of the features of a two material system it does not give a way to extract parameters from the results as was offered in the 1-D case. The analytical theory of the two-material case is currently being explored by the author. The difficulty with obtaining a closed form is that one requires two additional boundaries conditions at the material interface, x = 0. The first is trivial and comes from conservation of energy, for any real infinitesimal positive ϵ ,

$$k_1 \frac{\partial U(-\epsilon)}{\partial x} = k_2 \frac{\partial U(\epsilon)}{\partial x}.$$
(7.50)

However, the difficulty lies in obtaining a relationship between $U(-\epsilon)$ and $U(\epsilon)$.



Figure 7.3. Numerical simulation results for time-harmonic excitation of composite samples of glass and copper. Shown are the spatial distributions in space at t = 1 (a and b) second after turning on the excitation and the the temporal profile at the surface x = 0 (c and d). In each plot the blue line represents the composite (stack) results, the green line the results for copper and the red line the results for glass (reproduced from fig. 7.1 and shown here only for comparison).

Current work is being conducted on this topic. However, the form of the solution is assumed to be the same as the 1-D case that is,

$$u(0,t) = DC(P) + M(P)cos(\omega t - \eta(P))$$

$$(7.51)$$

where P is the set of parameters defined as $P\{L_1, L_2, D_1, D_2, k_1, k_2, \omega, h\}$.

Due to the difficulty of obtaining an analytical results, this system can be investigated following in the footsteps of the optical tomography and diffuse optical imaging fields. That is, to first simulate the data for a multi-dimensional parameter space, defining a look-up table, and then solve the inverse problem given a measurement of the surface magnitude and phase. In our case the parameter space is P. The look-up table parameter space can be restricted given a priori knowledge such as length of samples, excitation frequency, and material parameters. While solution uniqueness and computation complexity become issues, in principal the set P can be inversely determined from a surface measurement.

7.4 Experiments

7.4.1 Experimental setup

The proposed experimental setup is as follows. A MIRCat(Daylight Solutions) MIR laser is used to illuminate the sample of interest. The laser is intensity modulated by an optical chopper. A MIR polarizer (Thorlabs) is used in order to block any contribution of the signal from diffusely reflected light; this unfortunately blocks half of thermal light as well. To measure the signal the VOx microbolometer characterized in the beginning portion of the report is used. Since optical power cannot be negative the excitation,

$$\phi_{left} = A(1 + \Re(e^{i\omega t})) \tag{7.52}$$



Figure 7.4. Schematic of experimental setup. A,B,C,and D represent the optical chopper, sample, polarizer, and detector respectively.

and therefore the solution is a linear combination of the DC and harmonic solutions previously shown,

$$u(x,t) = mx + b + \Re(c_2 e^{-\sqrt{\frac{\omega}{2D}}x} e^{i(\omega t - \sqrt{\frac{\omega}{2D}}x)})$$

$$(7.53)$$

$$=\frac{-A}{hL-2k}x + \frac{A(k+hL)}{2hk+h^2L}$$
(7.54)

$$+\frac{A}{k}\sqrt{\frac{D}{\omega}}\cos(\omega t - \sqrt{\frac{\omega}{2D}x} - \pi/4).$$
(7.55)

Since the surface of the object will be viewed by the camera, only u(0,t) is measurable.

$$u(0,t) = \frac{A(k+hL)}{2hk+h^{2}L} + \frac{A}{k}\sqrt{\frac{D}{\omega}}\cos(\omega t - \pi/4).$$
 (7.56)

This method cannot alone determine the values of D and k, but rather the value \sqrt{D}/k . In addition since A will depend on the absorption coefficient, it may also be an unknown. While this limits the capability of this method for characterization, it is not an issue for many imaging application because this quantity will be different for different materials and can be used to discriminate them.

The power for this experiment is set to $\approx 400 \ mW$ on a 2 mm Gaussian spot



Figure 7.5. Temperature signal relative to glass with a thin layer of graphite. Showing the doubling of thermal signal.

size $(1/e^2 point)$. It is assumed that near the spot center the problem will still show characteristics of the 1-D analysis; this turns out to be the case. In analyzing the data the DC component will be subtracted and the magnitude of the oscillations will be determined which is proportional to \sqrt{D}/k . ω will be held at 44 rad/s. In order to fix the absorption coefficient for all the samples, each sample is coated with a thin layer of graphite-based dry lubricant. This material is used ubiquitously in the laser flash characterization method as it has very high absorption and its thermal conductivity and diffusivity are so high that it imperceptibly alters the measurement. Fig. 7.5 shows that a thin laser of graphite doubles the signal relative to the bare glass case. This also allows the method to be applied to reflective and semi-transparent materials.

7.4.2 Results

For the results all temperature measurements will be measured relative to that of glass. Therefore, a value of 2 "normalized temperature" means that,

$$2\frac{\sqrt{D_{measured}}}{k_{measured}} = \frac{\sqrt{D_{glass}}}{k_{glass}}.$$
(7.57)

Additionally, the data will be subjected to a 2 Hz digital bandpass filter centered around 7 Hz. Fig. 7.6 shows the simulated and experimental results for the largest signal measured, that of glass on aluminum. The plot also includes glass for reference (the magnitude of the glass oscillations is by definition 1). While the enhancement in the simulation predicts a factor of 11, the enhancement measured is seen to be a factor of 7 (see fig. 7.7). This could be due to a number of factors, but there are three likely candidates: first, there exists a range of possible values for different glasses and metals and a reference would be needed to have a more accurate result; second, the model did not include any interface resistance; and third, deviation of the measurement from a 1-D problem allowing heat to escape laterally.



Figure 7.6. (a) Numerical results of glass and glass on aluminum. (b) Experimental results glass on aluminum. Both are normalized such that the bare glass signal has an amplitude of 1.

In comparing the normalized temperature oscillations between different materials, the root-mean-square value will be used. This is opposed to directly comparing their frequency magnitudes as obtained via fourier-transforms (FTs). The RMS is chosen because it was found that the relative error in RMS is lower than that obtained via FTs. The FTs are less accurate due to frequency jitter in the chopper and timing jitter in the image acquisition time. The experiment was repeated 4 times glass(reference), aluminum, glass on aluminum(G on A), and aluminum on glass(A on G). These results are prestend in 7.7 where the blue dots represent the simulated results and the orange stars the experimental. The experimental results show excellent agreement with that of the simulated results. Further analysis, would require knowledge of the ground-truth values of the various D's and k's.



Figure 7.7. Relative amplitude coefficient(temperature). Blue stars are simulation and orange dots are experiments.

7.5 Proposed future work

7.5.1 Diffuse thermal tomography

Diffuse optical imaging (DOI) has been a wonderful technique developed in order to image inside of living tissue [9, 45]. The principle behind DOI is illustrated in fig. 7.8. A modulated source is incident upon a tissue sample; the photons penetrate into the tissue and are deflected, scattered, and absorbed as shown in fig. 7.9. If one is not interested in photons directly under the source, this is in practice always true, this problem can be treated as a diffusion problem, abiding by Fick's law [40]. The detector is placed a distance ρ away from the point of excitation on the surface of the object and measures a signal that has been attenuated and phase shifted according to the material's composition. For a given ρ the major signal contributions come from a banana shaped region. If there is an occlusion (a highly absorbing or scattering material) one can detect this by either changes in the magnitude or phase measured at the detector. Moving the detector allows one to reconstruct tomographic images.

It is readily apparent that this method is applicable to thermal imaging as well. In fact due to the diffusion approximation taken in DOI the problems are mathematically very similar. In order to extend 1-D thermal imaging shown in this paper to the 3-D dimensional case more analysis would have to be done. However, it should be noted that a closed form solution would not have to be obtained. In much of diffuse optics works in by simulating forward models and calculating the properties via inverse models. The same techniques are applicable to thermal diffuse imaging as well.



Figure 7.8. (a) Schematic of diffuse optical imaging. A modulated light source is incident on the material and a detector is place a distance ρ from the point of excitation. (b) At the detector one can see that the light is attenuated and phase-shifted relative to the input signal. Reproduced from [40].



Figure 7.9. Illustration showing that the majority of signal for a given source-detector separation comes from a banana shaped region(shaded). Changing the source-detector separation allows one to adjust the height of this region.

7.6 Conclusion to part II

In this second part the phasor solution to the 1-D homogenous equation was initially derived. A implicit numerical scheme was then outlined that used the general heat equation allowing for more general diffusivity profiles. The numerical method for the 1-D case matched exactly that of the numerical case. In addition it was shown that in the two material case the surface temperature oscillation amplitude can be amplified relative to the homogenous case dependent on material composition.

A laboratory setup was constructed to test this model. The setup utilized a MIR laser for the source, an optical chopper to modulate the light, a polarizer to block all reflected light, and the VOx microbolometer detector characterized in part 1 of this report. The results match well with that of the numerical results showing in fact an amplitude enhancement in the case of thermal insulator on a thermal conductor, specifically that glass on aluminum. The enhancement, however, was 7 instead of the predicted 11. The errors in this probably arise from three sources: incorrect numerical assumptions for the value of k an D, limitations of the 1-D method for a fundamentally 3-D problem, and the that the model does not include interface resistance.

Finally, it was shown that this measurement bares a striking resemblance to the measurement scenario that has lead to the lucrative field of diffuse optical imaging. It is proposed for future work that this parallel be investigated further to realize diffuse thermal tomography.

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