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FY-17 Report for the Design of a Benchtop PTR System

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1. Introduction

Photothermal Radiometry (PTR) is a versatile thermal property measurement technique based on the measurement of thermal radiation emitted from the sample. Its utility comes from its ability to perform noncontact thermal property measurements of layered or homogenous structures without the use of a transducer layer, as is necessary in thermoreflectance measurements. PTR measurements are conceptually similar to the standard laser flash technique, however, PTR measurements can be conducted with the heating and temperature detection on the same side of the sample. This is a significant advantage when samples are irregularly shaped, thermally thick or only one surface is prepared for measurement.

In general, PTR measurements use an intensity modulated laser to thermally excite a sample. The sample temperature response to the thermal excitation is observed by measuring the emitted thermal radiation. The laser modulation is generally either pulsed or periodic. For the scope of this work, only the periodic modulation will be discussed. The thermal radiation is collected through the use of reflective or refractive optics and focused to an IR detector. The alignment of the collection optics determine the area and location on the sample surface that the radiation is collected from.

This report provides a summary of the current design of a benchtop PTR system that will be used to develop capability to adapt the system to in-pile measurement of thermal properties. Preliminary modeling and experimental results are provided and a description of the path forward is provided.

2. Current Design of Benchtop System

2.1 Measurement Methodology

The measurement methodology employed in this study involves scanning the pump beam across the area on the sample that is imaged onto the IR detector (probe footprint). The output of the IR detector is recorded using a lock-in amplifier. As the separation distance between the pump and probe is increased, the phase lag of the temperature field at the probe position increases. Theoretical phase profiles recorded at several different frequencies are shown in Figure 1. Near the center of the scan (r=0), the phase profiles are dominated by the finite size of the pump and probe. At larger distances, the profiles become linear and are related to the thermal diffusivity.[a] The thermal diffusivity can be obtain by measuring the slope and calculating the diffusivity using:

$$slope = \sqrt{i\omega/D}$$

where ω and D are the angular frequency and thermal diffusivity respectively. A more accurate method is to fit both the spot size and the diffusivity to a continuum based model.[i]

[a] This is strictly true for uncoated substrates. A thin layer of carbon is applied to optically transparent substrates to ensure strong optical absorption. However, at the frequencies of interest for PTR systems (0.1 to 10 Hz), the influence of the carbon layer can be safely neglected.

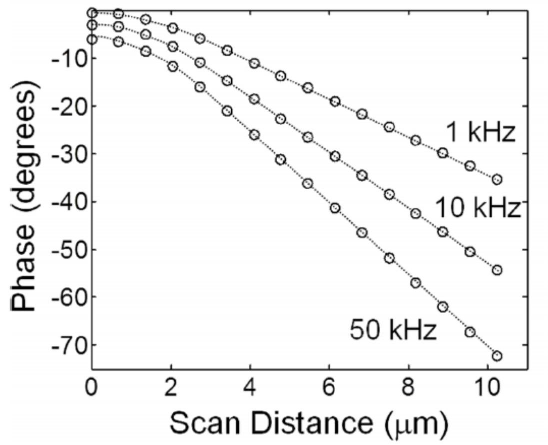

Figure 1. Thermal wave phase resonse. Far removed from the r=0, the phase profile is linear and related to the thermal diffusivity.

2.2 System Description

A simple benchtop PTR system has been designed and assembled. This system will allow various configurations and components to be tested and theoretical models to be validated. These investigations will support the development of a PTR system that is compatible with the in-pile measurement objective. Excitation for the benchtop PTR system consists of the beam from a 532nm free space laser. The beam passes through a mechanical chopper used for modulation and is then coupled into an optical fiber. The beam emitted from the distal end of the fiber is focused through a lens onto the sample surface. The fiber and focusing lens are mounted to orthogonal stages that allow the pump spot to be scanned in the horizontal and vertical directions. A schematic of the system from the fiber output to the detector is shown in Figure 2. The IR radiation from the sample surface is collected and collimated by a ZnSe lens place one focal length from the sample surface. The collimated radiation passes through a filter which transmits radiation in the 3-12μm wavelength range. The radiation is then focused by a second ZnSe lens onto the detector. The lenses both have the same focal length resulting in a 1:1 ratio of detector size to probe spot size on the sample. A photograph of the PTR system is shown in Figure 3. The pump beam and focusing lens, the sample, collection lenses, and detector are all mounted on manual stages to assist in alignment of the system.

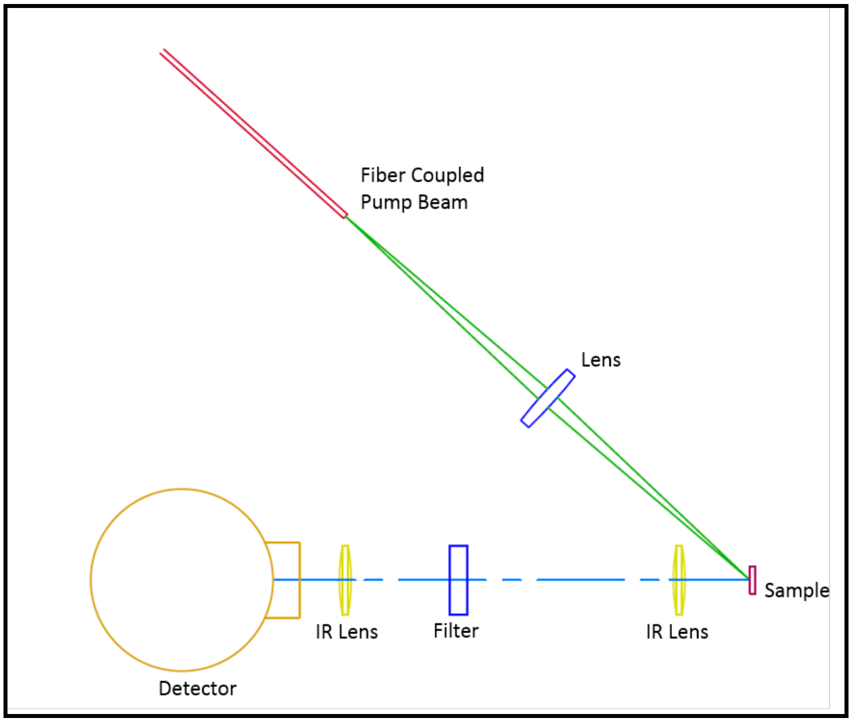

Figure 2. Diagram of current PTR benchtop system.

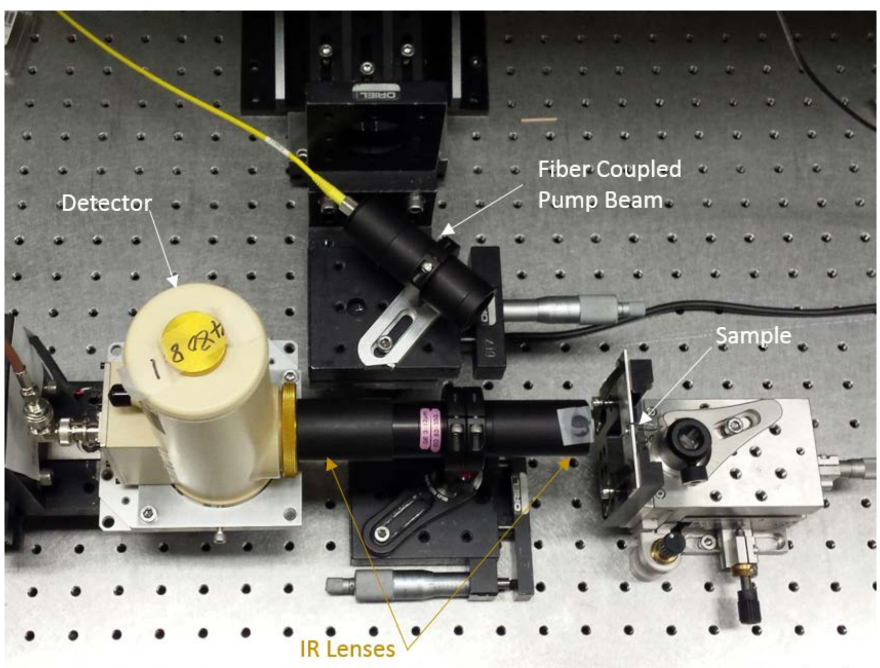

Figure 3. Photograph of current bench top system with labeled components.

Measurements made using the PTR setup are shown in Figure 4. Three data sets are shown which illustrate the importance of proper system set up. Each data set is from a different sample material, so the actual slopes are not important. Plot “a” shows data from a measurement scan with the sample out of focus. Note the shoulders on the slope of the phase. Plot “b” shows data from a scan with no aperture in the optical collection train. Note the slope flattens in the wings, possibly due to spherical aberrations. Plot "c" shows a scan with the proper shape. Note the near linear slope on both left and right sides.

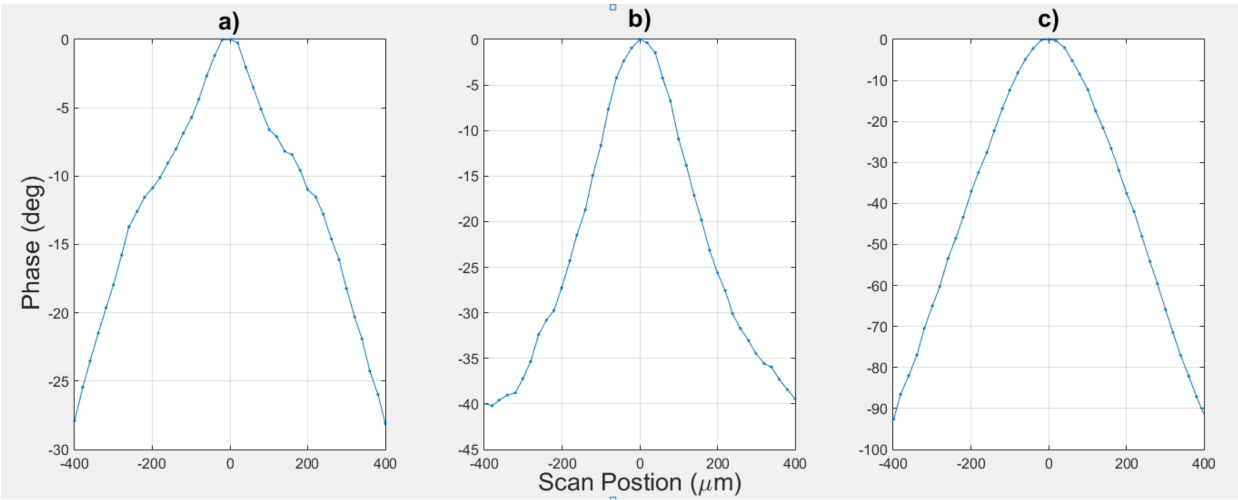

Figure 4. Preliminary PTR results showing the progression of improved measurement results.

3. Modeling Part One

Modelling of the expected response of a PTR system was initiated early in the project to support setup design. An initial modelling objective is to gain a better understanding of the differences between a reflected Gaussian probe beam commonly found in a thermoreflectance technique and an emitted probe spot as found in a PTR system. The measured temperature field at a point on the sample is given as a convolution between the spatial temperature field and the spatial distribution of the probe spot at the sample surface. For comparison, in the thermoreflectance approach, the probe spatial profile is characterized by the diameter of the probe beam at the sample surface. In PTR, on the other hand, the effective probe size is primarily determined by an area over which emitted IR radiation is collected on the detector. This area is represented by an image of the detector aperture on the surface of the sample. Depending on the optical configuration the measurement spot size can be significantly larger than the excitation spot size. In the initial modelling work, the measured temperature profiles were calculated with variable probe spot size to capture aforementioned response. It is assumed that the probe spatial profile can be represented by a Gaussian distribution. Additionally, the measured signal is assumed to be linearly proportional to temperature, an assumption that is not necessarily true for large changes in temperature.

Figure 5 summarizes the results of this analysis. It assumes an excitation with a spot size of 1 µm and modulation frequency of 1 kHz. We observe that for the smallest detection spot size, the measured profile is closest to the actual temperature profile. For the largest spot size considered, the measured profile deviates significantly from the actual temperature profile. In the intermediate regime the slope of the measured profile agrees reasonably with the actual profile, but careful examination suggests a slightly steeper slope.

The path forward for modelling will involve: 1 - accounting for the nonlinear dependence of emitted irradiation on temperature and (2) developing an optical model of a measurement spot size based on the provided configuration of optical elements.

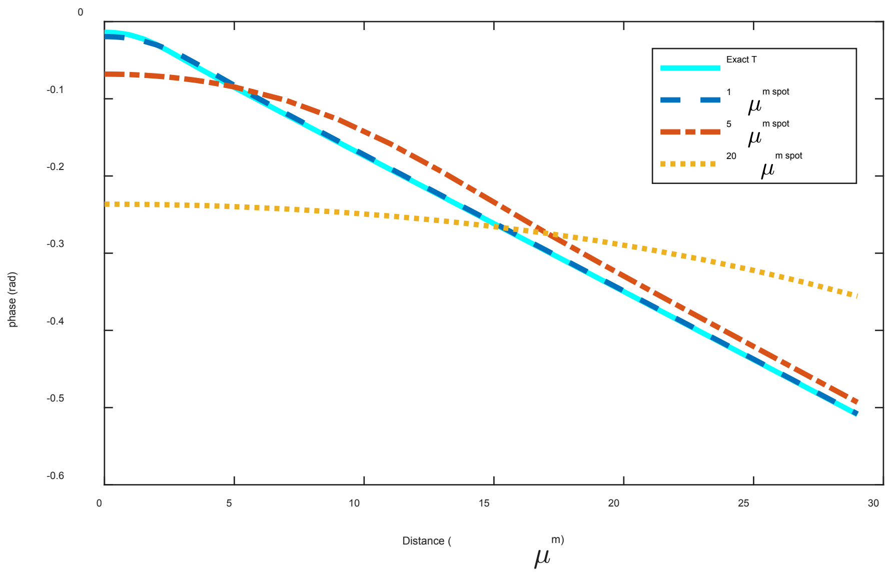

Figure 5. Comparison of profiles measured under different effective spot size.

4. Modeling Part Two

This modeling task involves identifying the appropriate length scales for measurement of bulk thermal conductivity in porous friable ceramic samples using PTR. As a starting point, porous samples were considered that are representative of fuels at various stages of burnup. To facilitate the discussion, the mean pore diameter is assumed to be 10 µm. This pore size distribution would suggest bulk-like behavior for measurement volumes larger than 100 µm. Additionally it is assumed that the smallest sample dimension is on the order of 1-2 mm and thus to avoid having to consider lateral and backside boundary conditions the maximum measurement volume should be less than 1 mm.

There are three length scales associated with the proposed measurement method. The first is the spot size of the pump beam at the sample surface. In light of the discussion above, the spot size should be equal to or larger than 100 µm. The other two length scales are the scan distance and the thermal wavelength. In order to avoid having to consider lateral and backside boundary conditions, these should be kept smaller than 1 mm. The modulation frequency of the pump laser must be chosen appropriately to keep the thermal wavelength within the 100µm – 1mm range. Using a thermal diffusivity value of 2.0×10^{-6} m^2/s, the modulation frequency range should be in the range of 0.75 Hz to 50 Hz.

To further explore the influence of various length scales we have reproduced a simple theory used to model the thermal wave response in an unbounded half-space.[ii] The pump laser is assumed to have a Gaussian profile and the laser energy is deposited within a few optical skin depths of the surface of the sample. The phase and amplitude profile for a 100 µm spot size are given in the upper panes of Figure 6. The scan distance has been nondimensionalized using the spot size. The three modulation frequencies considered were 0.75 Hz, 2 Hz and 50 Hz. The ratio of the thermal wavelength to the spot size is indicated for each frequency considered. The amplitude profile extends past the excitation spot for all of the frequencies considered. A similar presentation is given for a spot size of 500 µm in the bottom panes of Figure 6. A magnified view of the amplitude profile is shown in the bottom right pane of Figure 6. In this case the thermal wave amplitude extends past the excitation spot for only the lowest frequency considered. For the highest two frequencies, the thermal wave can only be measured with confidence within the excitation region. Unfortunately, in this region the phase profile is determined almost entirely by the spot size and has little dependence on the thermal diffusivity. Ideally to get an accurate estimate of the thermal diffusivity, the linear region of the phase profile should be well developed. It was found that this is the case when the ratio of μ to R is greater than 1. To meet this requirement, the smallest possible spot size should be used (~100 μm).

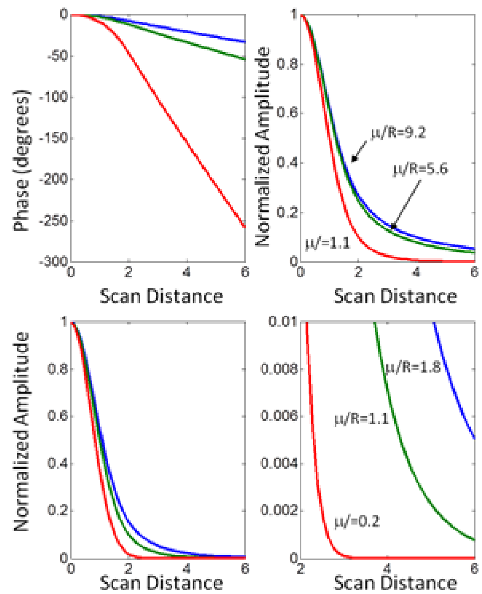

Figure 6. Top: Phase profile (left) and amplitude profile (right) for an excitation spot size of 100 μm. Bottom: Amplitude profiles for a 500 μm spot size. A magnified view of the amplitude profile is given in the bottom right. The spot size is used to nondimensionalize the scan distance.

5. Considerations for Future Design Optimization

Detection spot size, separation distance range, heating spot size, and modulation frequency are important factors that influence the measurement system capabilities. The heating spot size can easily be manipulated through the use of optical lens. Mechanical choppers, acousto-optical modulators, or electro-optical modulators are capable of modulating laser beams over a large frequency range. The main considerations for system configurations are the parameters that influence the alignment and detection spot size. These considerations are the focus of this section.

The collection optics play an important role in the experimental configuration of PTR systems. It is crucial to have a firm understanding of the detection spot size and location. There are several important practical considerations involved with the decisions associated with the collection optics. Spherical and chromatic aberrations can play a significant role in determining the detection spot size of the system. Additionally, the effective focal length of the lenses or mirrors used and the detector size determine the detections spot size of the system. These parameters needs to be chosen carefully in conjunction with the heating spot size, thermal diffusion length, and scanning distance. This section provides a discussion about collection optic decisions.

5.1 Reflective vs Refractive Optics

Optical components generally operate as a reflective or refractive element. Refractive optical elements manipulate light by refracting the light as it passes through the optical element. This refraction is governed by Snell's law

$$\frac{\sin(\theta_1)}{\sin(\theta_2)} = \frac{n_2}{n_1}$$

where n_1 and n_2 are the indices of refraction of the two materials, θ_1 and θ_2 are the angle of incidence and refraction. A materials index of refraction is a function of the wavelength of light. This wavelength dependence causes the performance of refractive optical elements to be a function of wavelength. Practical consequences result because the detection area on the sample surface is dependent on the light wavelength. These effects add difficulty in the alignment procedure and uncertainty in the detection area.

Reflective optical elements manipulate light by reflection off the surface of the element. Since reflection interactions may appropriately be assumed to be independent of wavelength, unlike refraction, reflective elements do not suffer from chromatic aberrations. Due to this fact, visible wavelength of light can be used for the system alignment since this light will travel along the same path as the IR, which facilitates reliable alignment of the system.

The current system design described previously is based on refractive optics. Near term testing of this configuration will likely include parameterized testing of aperaturing the detection lens to test for effects of spherical aberrations. Future modification of the refractive system may include optimizing these components (e.g. lens diameter, focal length) to achieve easier alignment for improved accuracy and to test for improved accuracy.

5.2 Photodetector Size

The selection of the photodetector size along with the optical line between sample and detector, determine the detection spot size. The size of the photodetector may be adapted to provide more ideal optical configuration depending on the desired probing size on the sample. Generally, the matching the detector size to the desired detection size has the potential to make alignment easier and reduce the complexity of the optical system required to resize the beam between the detector and sample. The performance of the detector is also related to its size. The detector noise is proportional to the square root of the detector area.

6. Summary and Conclusions

A benchtop spatial scanning PTR system has been designed and installed to allow for flexible optimization for measuring thermal properties of a sample. Preliminary experimental results show promising performance with potential performance improvements through system design optimization. Additionally, thermal wave models have been developed to support system design and measurement interpretation.

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[i] D. H. Hurley, R. S. Schley, M. Khafizov, B. L. Wendt, Local Measurement of Thermal Conductivity and Diffusivity, Rev. Sci. Instrum. **86**, 123901 (2015).

[ii] L. Fabbri and P. Fenici, Three Dimensional photothermal radiometry for the determination of the thermal diffusivity of solids, Rev. Sci. Instrum., 66 3593, 1995