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TRISO Fuel Thermal Conductivity Measurement Instrument Development

Colby Jensen
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TRISO FUEL COMPACT THERMAL CONDUCTIVITY MEASUREMENT INSTRUMENT DEVELOPMENT

by

Colby Jensen

A thesis submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

in

Mechanical Engineering

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2010
ABSTRACT

TRISO Fuel Compact Thermal Conductivity Measurement

Instrument Development

by

Colby B. Jensen, Master of Science
Utah State University, 2010

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Thermal conductivity is an important thermophysical property needed for effectively predicting fuel performance. As part of the Next Generation Nuclear Plant (NGNP) program, the thermal conductivity of tri-isotropic (TRISO) fuel needs to be measured over a temperature range characteristic of its usage. The composite nature of TRISO fuel requires that measurement be performed over the entire length of the compact in a non-destructive manner. No existing measurement system is capable of performing such a measurement.

A measurement system has been designed based on the steady-state, guarded-comparative-longitudinal heat flow technique. The system as currently designed is capable of measuring cylindrical samples with diameters ~12.3-mm (~0.5") with lengths ~25-mm (~1"). The system is currently operable in a temperature range of 400 K to 1100 K for materials with thermal conductivities on the order of 10 W/m/K to 70 W/m/K. The system has been designed, built, and tested. An uncertainty analysis for the determinate errors of the system has been performed finding a result of 5.5%. Finite element modeling of the system measurement
method has also been accomplished demonstrating optimal design, operating conditions, and associated bias error.

Measurements have been performed on three calibration/validation materials: SS304, 99.95% pure iron, and inconel 625. In addition, NGNP graphite with ZrO$_2$ particles and NGNP AGR-2 graphite matrix only, both in compact form, have been measured. Results from the SS304 sample show agreement of better than 3% for a 300–600°C temperature range. For iron between 100–600°C, the difference with published values is < 8% for all temperatures. The maximum difference from published data for inconel 625 is 5.8%, near 600°C. Both NGNP samples were measured from 100–800°C. All results are presented and discussed.

Finally, a discussion of ongoing work is included as well as a brief discussion of implementation under other operating conditions, including higher temperatures and adaptation for use in a glovebox or hot cell.

(94 pages)
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Colby Jensen
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<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>FEA</td>
<td>finite element analysis</td>
</tr>
<tr>
<td>GHP</td>
<td>guarded hot plate apparatus</td>
</tr>
<tr>
<td>HFM</td>
<td>heat flow meter apparatus</td>
</tr>
<tr>
<td>INL</td>
<td>Idaho National Laboratory</td>
</tr>
<tr>
<td>NGNP</td>
<td>Next Generation Nuclear Plant</td>
</tr>
<tr>
<td>ORNL</td>
<td>Oak Ridge National Laboratory</td>
</tr>
<tr>
<td>TFTCMS</td>
<td>TRISO fuel thermal conductivity measurement system</td>
</tr>
<tr>
<td>TRISO</td>
<td>tri-structural isotropic</td>
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</tbody>
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NOMENCLATURE

\( A_m \) \quad \text{Cross-sectional area of meter bars, } A_m = \pi r_b^2, \text{ [m}^2\text{]}

\( A_s \) \quad \text{Cross-sectional area of test sample } A_s = \pi r_s^2, \text{ [m}^2\text{]}

\( C_p \) \quad \text{Heat capacity of test sample, [J/kg]}

\( \frac{dT}{dz} \) \quad \text{Temperature gradient in the z-direction, [K/m]}

\( F_p \) \quad \text{Particle volume fraction factor}

\( h_b \) \quad \text{Height of meter bars, [m]}

\( h_s \) \quad \text{Height of test specimen, [m]}

\( k \) \quad \text{Thermal conductivity, [W/m/K]}

\( k_g \) \quad \text{Thermal conductivity of nickel guard as a function of temperature, [W/m/K]}

\( k_i \) \quad \text{Thermal conductivity of insulation as a function of temperature, [W/m/K]}

\( k_m \) \quad \text{Thermal conductivity of meter bar as a function of temperature, [W/m/K]}

\( k_s \) \quad \text{Thermal conductivity of test specimen, [W/m/K]}

\( k_{sc} \) \quad \text{Numerically calculated thermal conductivity of test specimen, [W/m/K]}

\( L \) \quad \text{Thickness of test sample, [m]}

\( \varphi \) \quad \text{Particle volume fraction}

\( q_1 \) \quad \text{Heat flow in hot-end meter bar, [W]}

\( q_2 \) \quad \text{Heat flow in cold-end meter bar, [W]}

\( q_s \) \quad \text{Heat flow in test sample, [W]}

\( r \) \quad \text{Unit vector in radial direction, [m]}

\( R_m \) \quad \text{Radius of meter bar, [m]}

\( R_g \) \quad \text{Radius of guard, [m]}
$R_i$ Outside radius of insulation, [m]

$R_s$ Radius of test specimen, [m]

$R_{th}$ Interfacial thermal resistance, [$m^2K/W$]

$t_{1/2}$ Time required for the rear face temperature to reach half of its maximum temperature, [s]

$T_{1-6}$ Monitored temperatures at positions $Z_{1-6}$, [K]

$T$ Generic temperature, [K]

$T_i$ Temperature along insulation boundary, [K]

$T_{ch}$ Sample stack temperature at the cold end, [K]

$T_{cg}$ Guard temperature at the cold end, $T_{cg}=T_{ch}+\Delta T_{bg}$, [K]

$T_{hb}$ Sample stack temperature at the hot end, [K]

$T_{hg}$ Guard temperature at the hot end, $T_{hg}=T_{hb}-\Delta T_{bg}$, [K]

$T_m$ Meter bar temperature at interface with test sample, [K]

$T_s$ Test sample temperature at interface with meter bars, [K]

$x_i$ A generic variable in uncertainty analysis

$z$ Unit vector in the axial direction, [m]

$z_{1-6}$ Monitor positions, “1” starting from the hot end and “6” ending at the cold end, [m]

$\bar{T}_1$ Average temperature of first meter bar, [K]

$\bar{T}_2$ Average temperature of second meter bar, [K]

$\bar{T}_s$ Average temperature of test sample, [K]

$\alpha_s$ Thermal diffusivity of test sample, [$m^2/s$]

$\Delta T_a$ Temperature deviation of the average guard temperature from the average specimen temperature, [K]

$\Delta T_{bg}$ Temperature difference between the meter bar and the guard, [K]

$\Delta T_{hc}$ Temperature difference between the hot and cold ends of the meter bars, $\Delta T_{hc}=T_{hb}-T_{ch}$, [K]
\( \Delta T_m \) Temperature difference between two temperature monitors on the meter bars, [K]

\( \Delta T_s \) Temperature difference from two temperature monitors on the test specimen, [K]

\( \Delta T_1 \) Measured temperature difference across first meter bar, [K]

\( \Delta T_2 \) Measured temperature difference across second meter bar, [K]

\( \Delta Z_1 \) Distance between temperature monitor positions in first meter bar, [m]

\( \Delta Z_2 \) Distance between temperature monitor positions in second meter bar, [m]

\( \Delta Z_m \) Distance between two temperature monitor positions on the meter bars, [m]

\( \Delta Z_s \) Distance between temperature measurement points in test sample, [m]

\( \rho_s \) Density of test sample, [kg/m\(^3\)]

\( \sigma_{A_m} \) Uncertainty in meter bar area

\( \sigma_{A_s} \) Uncertainty in test sample area

\( \sigma_{x_i} \) Uncertainty associated with variable \( x_i \)

\( \sigma_{\lambda_m} \) Uncertainty in meter bar thermal conductivity

\( \sigma_{\lambda_s} \) Uncertainty in test sample thermal conductivity

\( \sigma_{\Delta T_m} \) Uncertainty in temperature difference in the meter bars

\( \sigma_{\Delta T_s} \) Uncertainty in temperature difference in the test sample

\( \sigma_{\Delta Z_m} \) Uncertainty in the distance between thermocouples in the meter bars

\( \sigma_{\Delta Z_s} \) Uncertainty in the distance between thermocouples in the test sample
CHAPTER 1
INTRODUCTION

1.1. Background and Significance

As part of the development of advanced fuels and materials in the Next Generation Nuclear Plant (NGNP) program, a need exists for the capability to characterize the thermomechanical and thermophysical properties of these materials. An understanding of these properties is crucial for predictive capability and modeling and for characterizing the material behavior in both pre- and post-irradiation conditions. At times, the inherent composition and geometry in which these materials are used, as well as the environments in which they are to perform, requires specialized measurement tools.

One crucial thermophysical property needed for effectively predicting fuel performance is thermal conductivity. A method to measure the bulk, longitudinal thermal conductivity of tri-isotropic (TRISO) fuel compacts needs to be developed to provide for characterization and determination of changes that result in the irradiation of TRISO fuels. TRISO fuel is an NGNP fuel comprised of layered particles ~1-mm in diameter pressed and sintered together in a graphite matrix. The various layers of the particles serve multiple purposes including containing fission byproducts and maintaining the structural integrity of the particle.

Little data is available regarding the thermal conductivity of TRISO fuel. The only recorded data available is from German TRISO fuel work that is 20+ years old. Of course the German fuel used different graphite matrix materials than the NGNP TRISO fuel. Therefore, measured thermal conductivity of the NGNP TRISO fuel will be unique data and is an important part of the TRISO fuel development program.

For this project, TRISO fuel is in the form of a cylindrical compact measuring approximately 12.3-mm diameter × 25-mm length (0.5” diameter × 1.0” length). Because of the
composite nature of the sample, the bulk property must be measured on the whole compact. There is no existing measurement system available with the capability to do so in a non-destructive manner.

1.2. Thermal Conductivity Measurement

Thermal conductivity, which is the measure of a material’s ability to transport heat energy, is an intrinsic property of any material. It is defined as the quantity of heat energy transmitted per unit distance per unit temperature change over that distance in the direction of heat transfer. It is highly dependent on the chemical composition, physical structure, and state of the material. Because of its importance in characterizing material performance in nearly any engineering and/or science application, a vast amount of measurement methods and variations of those methods have been developed over the last 100+ years.

In spite of the number and variations that exist, these methods are generally classified as being transient or steady-state. Steady-state measurements depend on precise measurement of heat flow and temperature and a well-controlled pattern of heat flow. Transient measurements have many advantages over steady state, especially related measurement time and setup. Although simple in principle, steady-state measurements are generally quite complex in terms of systems and set up. Transient measurements, on the other hand, often use a more complexly derived thermal conductivity based on the set up of the method. Transient measurements usually do not require as much effort in terms of the setup and, relative to the steady-state type, are very quick to perform. In recent years, transient measurements have become much more common.

For the purpose of measuring the thermal conductivity of TRISO fuel compacts, available transient measurements methods are currently unable to perform the measurement needed due to the compact geometry and composition. For this reason, a steady-state measurement system based on the guarded-comparative-longitudinal heat flow technique has been designed, built, and
analyzed to investigate its use for measuring TRISO fuel compacts at high temperatures. The system has been used to measure the thermal conductivity of several samples including samples of known thermal conductivity for system validation and NGNP surrogate TRISO compacts.

Based on the results of the newly designed system, the lessons learned will lead to the development of a system with an expanded temperature range and with the potential to be used in a glovebox or hot cell where post-irradiation measurement of samples may take place.
CHAPTER 2

OBJECTIVES

The principal objective of this work is to non-destructively measure the bulk, longitudinal conductivity of NGNP TRISO fuel compacts. The TRISO fuel compact is a composite material with a nominal diameter of 12.3-mm (0.5”) and a length of 25-mm (1”). The desired temperature range for measurement is from 100 to 800°C. This overall goal may be broken down into smaller objectives as follows:

- Perform a literature review to select appropriate measurement method
- Design and construct a prototype system based on the literature review
- Perform initial testing of the system for calibration, by selecting and measuring samples of known thermal conductivity
- Measure surrogate NGNP TRISO compacts
- Perform an analysis of system response including measurement repeatability, uncertainty range, and bias uncertainty
- Perform a detailed finite element analysis (FEA) of the system design and operating parameters to aid in the understanding of system response and ideal operating conditions
- As part of longer term objectives, provide recommendations for implementing the selected method for other operating conditions including higher temperatures and use in a glovebox or hot-cell environment.
CHAPTER 3
LITERATURE REVIEW

3.1. Thermal Conductivity Measurement Methods

In literature, the methods and variations of methods of thermal conductivity measurement are numerous. Over the last century the primary methods of measurement used have changed, especially in the latter part of the century with the addition of new technologies. The point of this review is to provide a rather brief overview of the most relevant techniques as well as those that are currently more commonly used. The main factors contributing to the selection of a particular measurement method for solid materials are as follows [1]:

- Expected thermal conductivity of the sample
- Size and geometry
- Temperature range
- Magnitude of temperature gradient
- Accuracy required
- Electrical conductivity of the sample
- Fabrication difficulties
- Measurement time
- Density and specific heat of the specimen (if known)
- Level of porosity
- Inhomogeneities in the material (e.g., composite materials).

3.1.1. Transient Methods vs. Steady-State Methods

Steady-state and transient methods are the two typical categorizations of thermal conductivity measurement methods. The former measurement type relies on a steady-state
temperature gradient in the sample; the latter relies on a dynamic temperature field. Because a
dynamic temperature field also relies on thermal properties other than thermal conductivity, such
as specific heat and thermal diffusivity, the transient methods may also yield these other
properties [2].

Typical characteristics of steady-state methods are as follows [3-4]:

• Long measurement times
• Complicated apparatus/controls to create desired heat flows
• Measurements taken at mean temperature between hot and cold end of sample
• Temperature measurements may be difficult due to contact resistances.

Characteristics of transient methods are:

• Short measurement times
• Simpler setups than steady-state
• Measurement temperature gradients are very small [5]
• Smaller sample sizes.

3.1.2. Absolute Methods vs. Comparative Methods

Test methods may be absolute or comparative in nature, meaning that absolute results are
not dependent upon comparison with another material of known thermal conductivity. For
absolute measurements, careful calibration is required using appropriate certified reference
materials similar in type to the sample to be measured. Comparative methods are usually less
accurate, typically rendering them less desirable, but they also may allow less calibration work
[3].

The following briefly describes standard test methods for obtaining thermal conductivity
as well as other closely related methods from the literature. This review is not to be considered
fully extensive or all-inclusive; as mentioned before, the variety and quantity of measurement
methods is extremely numerous. For such a review one should refer to References [6-13]. Standards from the American Society for Testing and Materials (ASTM) are presented first, followed by other more common methods for measuring thermal conductivity.

3.2. Transient Methods

In recent years transient thermal conductivity measurement methods have become increasingly popular due to the characteristics listed in Section 3.1.1. Method reviews are presented in no particular order.

3.2.1. Standard Test Methods

This section describes transient thermal conductivity measurement methods that are defined by ASTM standards.

3.2.1.1. Line Heat Source Methods

ASTM C 1113 - Standard Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique) [14]

ASTM Test Method C 1113 is intended for use with isotropic, non-carbonaceous, dielectric refractories. The hot wire is an absolute, transient, direct measurement method of thermal conductivity. Thermal conductivity measurements can be made from ambient to 1500°C on refractories with thermal conductivities of less than 15 W/m/K. Method C 1113 consists of a pure platinum wire placed between two specimen bricks. An electrical current is applied to the wire, and rate of temperature increase of the platinum wire is accurately calculated by measuring the change in resistivity of the wire. The rate of the temperature increase in the wire is dependent on the rate at which heat flows into the constant temperature brick that surrounds it. Thermal conductivity is calculated based on the rate of temperature increase of the wire and power input.

Four variations of the hot-wire method exist in literature, differing mainly in temperature measurement procedure, and have been used on a variety of materials such as ceramics, fluids,
and polymers. The hot-wire is a widely used method for many reasons: it is generally considered effective, accurate, and absolute; measurement is taken at a fixed temperature eliminating the “mean temperature” inherent to other methods because in this case, temperature gradients across the sample are very low; and like other transient methods, it is typically faster than steady-state methods [5].

Although Method C 1113 is for dielectric refractories, the hot-wire method has been used on other materials including electrically conductive materials [15]. Some literature reveals that results become more scattered for higher thermal conductivity measurements (>50 W/m/K).


This test method is similar to ASTM C 1113 and is for measuring the thermal conductivity of thermoplastics, thermosets, and rubbers, filled and reinforced, with thermal conductivities in the range from 0.08 W/m/K to 2.0 W/m/K over a temperature range of -40–400°C. In this method, a line source of heat is located at the center of the specimen being tested, which is at a constant initial temperature. A known amount of heat is then applied to the specimen through the line source, which is transmitted radially through it. The temperature rise over time of the line source is measured from which thermal conductivity may be calculated.

Although an attractive method, the hot-wire method will not work for the objectives of this project as it would require a custom sample. The line heat source would have to be embedded in the sample as well as be electrically insulated. The expected thermal conductivity range of the TRISO fuel compacts is slightly on the high side for this method as well.

3.2.1.2. Flash Method


The laser flash method is used to measure thermal diffusivity of homogeneous solid, opaque materials with thermal diffusivity values between 0.001 cm²/s and 10 cm²/s in the
temperature range of 75–2800 K. Testing is performed in a vacuum or inert gas environment (with the exception of room temperature tests) on circular disks with thicknesses of 1.5–4-mm and diameters of 6–18-mm. The flat specimen is heated on one side with a laser pulse, and the temperature of the other side is measured over time. The time required for the rear face temperature to reach half of its maximum temperature rise is used along with the specimen thickness to calculate the thermal diffusivity:

\[ \alpha_s = \frac{0.13879 L^2}{t_{1/2}} \]  

For thermal conductivity, knowledge of density and heat capacity must also be known. In some systems, specific heat capacity may also be measured [18]; thus, only density is required to calculate thermal conductivity from the fundamental relationship:

\[ k_s = \alpha_s \rho_s c_p \]  

The main limitation of this method applied to the current objective is the sample size and shape. The current test sample is too large and because the TRISO fuel pellets are made of particles approximately one millimeter in diameter, a sample that is in the appropriate thickness range may not accurately represent the bulk material. For composite materials, the recommended specimen size for any thermal conductivity measurement is twenty units in the measurement direction, where a unit is the thickness of the thickest slab, plate, or in our case, a TRISO particle [9].

3.2.1.3. Modulated Temperature Differential Scanning Calorimetry


Test Method E 1952 presents a procedure for measuring the thermal conductivity of homogenous, nonporous solid materials between 0.1 W/m/K to 1.0 W/m/K over the temperature range of 0–90°C. This technique involves applying an oscillatory temperature to two specimens
of varying thicknesses (one thin), which creates an oscillatory heat flow into or out of the specimens. Heat capacity of the specimen can be derived from the amplitude of the heat flow and the amplitude of the oscillatory temperature that creates it. The thermal conductivity can be calculated from the apparent heat capacity of the thicker specimen, the actual heat capacity of the thin specimen, and other geometric and experimental constants.

This method does not have the range of thermal conductivities or temperatures required for this project. Again the compact’s geometry will not work with this method. For these reasons, this method is not considered a viable option.

3.2.1.4. Thermal Capacitance Calorimeter


This method is for determination of thermal conductivity of solid materials in the range of 0.02 W/m/K to 2 W/m/K over the temperature range of 300–1100 K. It is particularly useful in testing materials that are both reactive and undergo significant dimensional changes at high temperatures. The thermal capacitance calorimeter consists of a thermally conductive slug surrounded by a specimen material of much lower thermal conductivity. The assembly is subjected to a temperature change that causes heat to flow through the specimen layer into or out of the slug. The temperature change of the slug is controlled by how much and the rate at which heat is conducted through the specimen material, its mass, and its specific heat capacity. Using these properties and temperature measurements of the slug, heat flux may be calculated. The temperature gradient across the specimen is also measured. Combined with the heat flux and geometric data, thermal conductivity may then be calculated.

The test method is not applicable for the design in this project because it requires custom sample geometry. Also, the expected thermal conductivity of the surrogate test compacts is too high to meet the requirements of this method.
3.2.2. **Non-Standard Test Methods**

This section describes several common transient thermal conductivity measurement methods that are not currently defined by ASTM standards.

3.2.2.1. Plane Source Methods

**Transient Hot-strip Method**

The hot-strip method [21-22] is a form of the plane source method. It is an absolute measurement closely related to the hot-wire method, but a long, thin strip is used as the heat source and temperature sensor instead of a wire. The strip is used as both the heat source as well as the temperature sensor where the resistance of the material is measured giving temperature. This method has the advantage over the hot-wire method that better thermal contact may be achieved with solid materials, whereas the hot-wire method is more limited to fluids and solids that can be cast around the wire [21]. Again the sample geometry eliminates this method as an option for measurement.

**Transient Hot-disc Method**

The transient hot-disc method is first described by Gustafsson [23] and is a specific case of the transient plane source technique. The hot-strip technique was actually a precursor to the hot-disc method. The hot-disc technique has been used to measure the thermal conductivity of materials, such as those with low electrical conductivity, anisotropic solids, building materials, stainless steel, thin metallic materials, and copper powder [24]. It can be used on materials with conductivities in the range of 0.005–500 W/m/K. The flexibility of this technique, including the wide range of temperatures, thermal conductivity values, and materials types from liquids, solids, and powders make this a very attractive method.

This method was not a good choice for this project as the high temperatures (~800°C) create problems in making a sensor to withstand them. The development of such a device would
also be excessively time intensive for this project. Due to the composite nature of TRISO fuel, another potential problem with this method is whether the measured thermal conductivity would be representative of the bulk material or a localized portion. For these reasons, this method was not selected.

3.2.2.2. “3-ω” Method

The 3-ω method [25] is a transient method very closely related to the hot-wire/hot-strip techniques in that it uses a single element as both a heater and a thermometer. The difference is that where the hot-wire technique measures temperature response with respect to time, the 3-ω method measures temperature response as a function of excitation frequency [25]. This method’s advantage is that it is insensitive to the errors from black-body radiation because the effective thickness of the sample is extremely small [26]. Borca-Tasciuc et al. has a good summary of the work that has been done using the 3-ω method [27]. This method can be used on any dielectric bulk solid or thin film with a smooth, flat surface about 1 cm × 0.5 cm.

Similar limiting factors as previously discussed methods prevent application of this method for this measurement. One is that it requires the specimen be electrically conductive or that a metal strip is artificially deposited on the surface to serve as the heater and the temperature sensor. Another is whether this method will work to capture the bulk property of the composite.

3.3. Steady-State Methods

At this point it is worth discussing some of the distinct characteristics in steady-state measurements. Four traditional classifications of steady-state measurements recognized are axial flow, radial flow, guarded hot plate, and direct electrical heating [8]. The first three could be more generally classified as unidirectional heat flow methods. The measurement principles are largely the same wherein the main objective is to create unidirectional heat flow in a sample with a
known heat flux. Measuring the temperature gradient, thermal conductivity may be calculated using Fourier’s basic relation in 1-D:

\[ q_s = -k_s A_s \frac{dT}{dz} \]  

(3.3)

For a homogenous material assuming constant area of conduction and constant thermal conductivity, this equation may be integrated between two endpoints and solved for the thermal conductivity of the material as:

\[ k_s = -\frac{q_s \Delta Z_s}{A_s \Delta T_s} \]  

(3.4)

The main difference of the methodologies of these three methods is due to the range of thermal conductivities of the sample materials that they were intended for and, to a lesser extent, the sample size.

3.3.1. Standard Test Methods

This section describes transient thermal conductivity measurement methods that are defined by ASTM standards.

3.3.1.1. Guarded Hot Plate


The guarded-hot-plate apparatus (GHP) is intended for use with flat, homogeneous specimen(s). It has been designed for use over a wide temperature range as well as for a wide variety of specimens. The GHP is a primary (or absolute) method. It can be operated in either double-sided mode or single-sided mode (ASTM C 1044).
The purpose of a GHP is to create measurable, unidirectional heat flow in a test specimen of known dimensions. The device consists of a guarded heater unit on one side of the test specimens, made of a concentric guard heater with a separately heated center metering area. On the other ends, the specimens are in contact with “cold plate” assemblies. For single-sided operation, another guard heater covers the meter plate opposite the specimen. The secondary guard is temperature controlled to help prevent lateral heat flows. Ideally, the guarded heater assembly and cold-plate assemblies have isothermal surfaces in contact with the test specimens and the temperature of the primary guard matches that of the meter plate. Heat flows unidirectionally from the meter plate into the cold plates. Thermal conductivity is calculated from Fourier’s Law based on the measurements of heat flow from the meter plate, measured temperature gradient across the specimen, metered section area, and specimen length.

This test method is generally used to measure materials of low thermal conductivity in the range of \( k < 1 \text{ W/m/K} \) [8]. Work has been done using GHP for testing samples with a characteristic size on the order of 1 cm [31] up to 1 m to temperatures over 1000°C [4]. Although this method will not be used for the current measurement design, much work has been done with GHPs in the last several decades [32-35]. Thus, many lessons learned from this work may be applied to the design of a system for measuring the TRISO fuel compact.

3.3.1.2. Heat Flow Meter Apparatus


The heat flow meter (HFM) has many similarities to C 177 and is widely used because of its simplicity and relatively quick measurement time. By this method, steady-state, one-dimensional heat flow is established through a specimen sandwiched between a hot plate and a cold plate. It also may utilize edge guards to control lateral heat flow. A heat flux transducer(s)
calibrated to standards is placed in the heat flow path to measure the heat flow rate. Test method C 518 has been used with temperatures up to 540°C. Uncertainty can be small with the HFM apparatus if calibration is performed with a material of similar thermal conductance, at similar thicknesses, mean temperatures, and temperature gradients as the test specimen. Thermal conductivity is calculated similar to C 177. Test Method E 1530 is very similar to C 518 but is modified to accommodate smaller test specimens, having an approximate thermal conductivity range of 0.1 W/m/K to 30 W/m/K over a temperature range of 150–600 K.

This test method as written is not ideal for the current objectives as the TRISO compacts are too small. As will be discussed later, the method chosen will use a similar idea as the heat flow meter but in a different form.

3.3.1.3. Comparative Axial Heat Flow Method


Test Method E 1225 is for materials with effective thermal conductivities between 0.2 W/m/K and 200 W/m/K over an approximate temperature range of 90–1300 K. This method falls under the category of an axial heat flow method. The main distinguishing point between this method and the guarded linear/Forbe’s Bar type (Section 3.3.2.1) is the manner in which the heat flow is measured. The latter incorporates a direct measurement of power to the sample heater while the former introduces a reference or comparator sample of known thermal conductivity from which the heat flowing through it may be calculated. Deducing the heat flow in this way introduces error associated with the value of the thermal conductivity of the reference sample.

This method was adapted for use in this project. Therefore, a more detailed description and review will follow in Section 3.4.
3.3.2. **Non-Standard Test Methods**

This section describes several common steady-state thermal conductivity measurement methods that are not currently defined by ASTM standards.

3.3.2.1. Absolute Axial Heat Flow Methods

Absolute axial heat flow methods are generally referred to as the guarded linear method or the Forbes’ Bar Method. These methods are not discussed much in literature beyond 1980 and were frequently used from about 1890–1975 [39] for highly conductive materials. Newer transient methods have made these methods obsolete in many circumstances.

These axial heat flow methods [39-40] are steady-state, absolute methods. Samples are generally long bars with length-to-diameter ratios on the order of about 10. For that reason, these methods are of particular interest to this project. As was previously mentioned, the overall concept is very similar to that of the GHP. The guarded linear method uses a heater/cooling system to establish a temperature gradient in the sample. A heated guard is used to prevent lateral heat losses from the sample. Measurement of the temperature gradient in the sample is taken, from which thermal conductivity is calculated with the heat input and sample length. Generally a tube “guard” with insulation between the tube and the sample is used. The Forbe’s Bar method differs from the guarded linear method in that precise matching of the linear tube guard temperature gradient to the sample temperature gradient is not necessary. Radial heat losses are estimated from sets of readings taken with two different guard temperature distributions. With these estimations, heat loss corrections are made to the measured value of heat flow in the sample.

The axial heat flow techniques are most suitable for small specimens with medium-to-large thermal conductivity values (k > 1 W/m K) [41] over a temperature range of T < 100 K up
to 1500 K. Additionally, they are very useful for the measurement of other properties such as electrical conductivity and thermoelectric power [42].

One of the main challenges presented in the literature related to this method is how to control heat leaks from the sample heater. Many concepts and principles of these methods are incorporated into our design, as the measurement setup is very similar, and these systems have been discussed extensively in literature [39-41].

3.3.2.2. Radial Heat Flow Method

The radial heat flow method [43] typically consists of a cylindrical sample which has a cylindrical heater located along its longitudinal axis. The centerline heater heats the center and heat dissipates through the sample to its outer surface. The sample must be long enough or the ends guarded in such a way that near mid length, the heat flow may be assumed to be only in the radial direction, meaning that only a radial temperature gradient may exist. Once steady-state conditions are achieved, temperature is measured at two known radial locations. From these temperatures and radial measurements, as well as the heat-per-unit length supplied to the heater, thermal conductivity may be calculated.

This method has a distinct advantage over many other steady-state methods: the need for complicated guarding systems may be eliminated. The obvious limitation for this method for the objectives of this project is sample geometry/thermal conductivity as well as the need for a non-destructive method. To use this method, a heater must be placed through the center of the sample and thermocouples embedded at different radial locations. The requirement that the sample measurement be non-destructive eliminates this method as an option.

3.4. TRISO Fuel Thermal Conductivity Measurement Method

Because of the cylindrical shape and medium-to-high thermal conductivity expected of the nuclear fuel to be measured, an axial heat flow method was selected. Further, due to the small
size of the sample and the desired temperature range for measurement, the comparative axial heat flow technique [44] was selected, as mentioned in Section 3.3.2.1.

The comparative axial heat flow method is a comparative, steady-state method. It has been used since the 1930s [45] and was more completely studied and developed in the 1950s and 60s by Ballard et al. [46], Morris and Hust [47], Franel and Kingery [48], and Mirkovich [49], among others. Laubitz [39] questioned the claimed accuracy of such measurements, but later studies performed by Sweet et al. [50] and Pillai and George [51] reported accuracies independent of the uncertainty of the reference sample, to be better than ±5%. In 1987, the ASTM produced a standard for this method, ASTM E1225 [38], which was revised in 2004. Also noteworthy for the purposes of this project, Babelot et al. performed tests on a modified commercial comparative thermal conductivity apparatus that was to be used in a glovebox [52], although information about their work is limited.

In this technique, a test sample of unknown thermal conductivity, $k_s$, is sandwiched between two reference samples of known thermal conductivity, $k_m$, making up what is termed the sample column or test stack. A temperature gradient is set up through the test stack such that it may be measured in each of the three samples. From the measured gradients in the reference samples and the cross-sectional area of the reference samples, the heat flowing through them may be calculated.

$$q_{1,2} = k_m \langle T_{1,2} \rangle A_m \frac{\Delta T_{1,2}}{\Delta Z_{1,2}}$$

Thus the reference samples act much like heat flow meters and are often referred to as meter bars. Using the averaged measured flux in the both meter bars and the measured temperature gradient in the test sample, the thermal conductivity of the test sample at its average temperature may be calculated.
Limited historical data is available for the thermal conductivity of the graphite materials used in TRISO-coated fuels developed in Germany in the 1980s. Gontard and Nabielek [53] report empirical values of thermal conductivities of two graphite matrix-only types cured at two temperatures as shown in Figure 3-1. Information is limited regarding the origin and acquisition of the graphite matrix data and the methodologies used to come up with the particle volume effect. This data is for the graphite matrix material alone ($\varphi = 0$) showing a range of 65–25 W/m/K over a temperature range of 100–1000°C. The effect of particle volume loading fraction, $\varphi$, is given in the report as a multiplying factor, $F_p$:

$$F_p = \frac{1 - \varphi}{1 + \varphi/2}$$

(3.7)
The consequence of the particle volume loading fraction is shown in Figure 3-2 for the A3-27 1800°C graphite from Figure 3-1. The expected particle volume loading fraction for the current TRISO fuel compacts to be tested is in the range of 0.3 to 0.38. For a value of 0.3, the value of thermal conductivity for the fuel is ~0.6 of the matrix-only material.

This legacy data provides a range estimation of possible thermal conductivity values and was considered in the design of our measurement system. Because the graphite material is different in NGNP fuels as well as having a higher particle volume fraction, their thermal conductivity values are expected to differ from the German data.

Figure 3-2. Thermal conductivity data taken from a German report [53] from the 1980s for A3-27 1800°C graphite. The particle volume fraction (φ) = 0.3, 0.35, 0.4 data represents an expected range for the thermal conductivity of the samples to be measured in this project.
4.1. System Design

Much of the current design for the TRISO fuel thermal conductivity measurement system (TFTCMS) is adapted from the guidelines given in ASTM E 1225-04 [38] as well as various pieces of relevant literature. The experimental environment is capable of a temperature range of 20–900°C, although the immediate objective was to test up to 800°C. This design summary is divided into sections about the main environmental control and data collection system and what is termed the core experimental measurement section.

4.1.1. Main System Overview

The environment required for this test needs the following features: controllable, steady temperature range of 100–800°C; gas tight; capability to operate in an inert atmosphere; and some means of passing instrumentation wiring in/out of the gas tight seals. Figure 4-1 shows an overview schematic of the primary environmental and instrumentation systems for the measurement. A 76-mm (3") quartz tube (MTI OTF-1200X) horizontal tube furnace capable of steady-state operation to 1100°C serves many purposes. The measurement section is placed into the tube furnace, as shown in Figure 4-1. Most importantly, the furnace provides the ambient temperature of the measurement in the area of the sample. The measurement setup also takes advantage of the natural temperature gradients created in the furnace, which assists in creating the temperature gradient controlled within the core measurement section, hereby reducing the power requirements of the control heaters (discussed more in Section 4.1.2).
Figure 4-1. Schematic overview of major system components showing system setup.

The system is also capable of operation under vacuum or an inert gas atmosphere. Vacuum is generally not recommended for this type of measurement [38, 40], as it can increase thermal contact resistances, especially at low temperatures. Therefore, a vacuum pump is used only to purge air from the system before introducing the inert gas, high-purity helium. Helium was chosen to provide the inert environment because it has a significantly higher thermal conductivity than any other inert gas. This property of helium helps to reduce contact resistance [4], which is especially important at lower temperatures. At higher temperatures, radiation heat transfer between the adjoining surfaces becomes increasingly dominant and helps overcome contact resistance issues between components. The inlet flow of helium is controlled by a float flow meter. In order to ensure no leakage of air into the system, a positive pressure of ~5-7.5-cm (~2–3") of water is maintained within the measurement tube. A very small and constant flow of helium is maintained through the system to provide for this. After initial testing, a gas purifier will be used to more fully ensure an inert atmosphere.

All instrumentation wiring passes through a four-way cross with metal gaskets at the outlets. The gaskets have been modified to allow the wiring to pass through while maintaining a
tight seal. The control instrumentation of the system consists of a dual-loop temperature controller (Eurotherm 3504) and a power supply (TDK-Lambda) to create and maintain temperature gradients in the sample column region as well as the guard (discussed more in Section 4.1.2). All measurement thermocouples are connected to a data acquisition unit (Agilent 34970a). A computer is used to record all data as well as to control the controllers of both the experimental section heaters and the furnace, allowing for nearly complete computerized control of the system.

4.1.2. Core Measurement Section Design

Figure 4-2 shows a schematic of the core measurement section of the TFTCMS. Work performed by Didion [54] that gives some basic guidelines for designing this type of measurement system assisted much of the sizing and choice of materials used in the design as well as ASTM E 1225-04.

The critical functions of the design of the experimental section are:

1. Create a controlled, one-dimensional, steady-state temperature gradient through the sample column (test sample and adjacent reference samples). This is accomplished through the use of a surrounding layer of insulation as well as a guard tube that will be matched closely to the temperature gradient in the central column.

2. Measure steady-state temperature gradients in the experimental sample and reference samples from which thermal conductivity may be calculated. Also, additional temperature measurements along the experimental region may be used to help account for any radial losses that could possibly occur.

3. Create reproducible conditions in the measurement region by use of a spring system to apply a desired pressure through the central column, creating reproducible contact resistances at the interfaces of the experimental sample and reference samples.
Figure 4-2. Schematic of the core experimental TRISO fuel thermal conductivity measurement system.
ASTM E 1225 recommends that the meter bars have a similar conductance to that of the sample [38]. Because the expected thermal conductivity of the nuclear fuel samples to be tested was in the 20–40 W/m/K range, a reference material with a similar conductivity was sought. Stainless steel 304 was selected for this reason (see Figure 6-1) and because of the numerous recommendations [50, 55-56] in literature as a material with a very low scatter of data among many sources [57-59]. The meter bar on the hot end was designed to hold a cartridge heater inserted in the end opposite of the test sample, which provides control of the sample column temperature gradient. The meter bar was made long enough to provide space between the heater and the measurement section of the meter bar to allow for a more uniform heat flux to develop.

One feature of this apparatus that greatly simplified its design and operation is a radiative-type heat sink used to dissipate heat away from the sample column and guard. The heat sink basically consists of a solid cylinder made of commercially available nickel alloy 201, chosen based on the same criteria discussed by Flynn [32]. Nickel has a relatively high thermal conductivity (~90 W/m/K for elemental nickel and ~ 60 W/m/K for nickel alloy 201), is resistant to oxidation, and is also relatively inexpensive compared to other candidate materials discussed by Flynn such as gold, silver electroplated with gold or nickel, and copper electroplated with gold or nickel. The heat sink is connected to the guard and one of the meter bars with threads to provide good thermal contact and extends out of the hot zone of the quartz tube furnace. By moving farther in or out of the furnace, the amount of heat it radiatively dissipates decreases or increases, allowing for more control of the sample temperature gradient.

As mentioned previously, the system is designed so a reproducible and constant force is created through the sample column to ensure good contact at sample interfaces. A stainless steel spring is supported by stainless steel rods that extend outside the heated zone to a nearly ambient temperature zone. In this way the spring force remains constant as it does not experience much of a temperature change.
A guard tube surrounds the sample and is filled with powder insulation. Diatomaceous earth powder was selected as the insulator for its good insulative properties and because it is readily available and will fill in around all components to prevent any unwanted heat flows. A custom-made tubular heater is wrapped around the guard at approximately the same axial location as the heater placed in the meter bar. Both heaters are controlled by a separate controller to create the desired temperature gradient in the apparatus.

Temperature is measured from Type N thermocouples mounted on the sample column and guard. Type N thermocouples were selected over Type K because they have greater stability when exposed to high temperatures [60]. Platinum-type thermocouples were considered and may be experimented with, but due to their high cost will not be used extensively. Initial testing (results presented later) used 0.076-mm (0.003”) Type N thermocouples. Due to their delicateness and the fact that high-temperature thermocouple drift has more effect with decreasing thermocouple size, they have since been changed to 0.127-mm (0.005”) Type N thermocouples. Size of the thermocouple should be kept small as to avoid creating heat paths that will disturb the temperature profile on the sample in the locations they are installed. Additionally as is discussed in Section 5.1, the larger thermocouple size contributes to the error in the measurement as well. Thermocouple wire is insulated using 1.587-mm (1/16”) Nextel 312™ ceramic fiber sleeving. The thermocouples were joined to the surface of the samples in small grooves using Omega brand CC High Temperature Cement. Thermocouple drift especially related to contamination [40] is still a major concern with this system, and a solution for this problem is unknown. This will require that measured EMF be monitored to make sure the readings do not drift to the point of causing problems. When such drift occurs at high temperatures, the thermocouples must be replaced.
4.2. Measurement Procedure

The following section discussed the setup and actual measurement processes used to obtain a thermal conductivity measurement at a particular temperature. The processes are broken down into assembly, running, and calculation.

4.2.1. Assembly Procedure

The custom samples used for the calibration tests are machined to the approximate size of a fuel compact with a length of 25-mm (~1") and a diameter of 12.3-mm (~0.5"). Two small grooves are made on the surface, perpendicular to the sample axis. The approximate locations of the grooves are 2.5-mm (0.1") from each of the end surfaces, giving an approximate separation of about 20-mm (0.8") between each groove. Similarly, in the meter bars, grooves are placed 2.5-mm (0.1") from the surface that contacts the test sample, and a second groove is placed approximately ~20-mm (0.8") from the first.

Thermocouples are assembled using 0.076-mm (0.003") and later 0.127-mm (0.005") Type N thermocouple wire. Nextel 312 sleeving is used to insulate each wire strand with a piece of 1.587-mm (1/16") mullite thermocouple tubing at the tip where the thermocouple bead comes together. Because the NGNP graphite test samples cannot be drilled or modified, the thermocouple bead is placed on the circumferential surface at locations similar to what was previously described. A 0.254-mm (0.01") nichrome wire is wrapped around the sample directly on top of the bead and tightened to ensure good contact between the bead and the sample, as seen in Figure 4-3 for an NGNP composite graphite compact. After bead separation distance is measured (following paragraph), the bead is then coated with Omega CC High Temperature Cement to also help ensure good thermal contact and hold it in place.

Before the bead is coated with cement, measurements are made on the thermocouple locations on each of the three pieces that make up the sample column. The procedure for
measurement of bead separation distance follows. The overall length of each meter bar and the test sample is measured using a micrometer or caliper. A Canon T1i 15.1 megapixel camera with a Canon EF-S 60-mm f/2.8 Macro lens was then used to take a close-up photograph of each with the thermocouple bead locations exposed (see Figure 4-3 for an example). These pictures were then imported into Matlab where a custom program was used to calculate the fraction of the overall length of the each piece that the distance between the thermocouple beads makes. The resolution of this camera allows for approximately: 0.0169-mm per pixel on a ~76-mm (~3”) sample and 0.00673-mm per pixel on a ~25-mm (~1”) sample. Based on these resolutions, the overall dominant source of uncertainty in the measurement is the size of the thermocouple bead, or about twice the diameter of the wire used (discussed more in Section 5.1).

After the thermocouples are bonded into place on the meter bars and the test sample, assembly of the entire core measurement section begins. The sample column comprised of the two meter bars and the sample is stacked. A 12.7-µm (0.0005”) piece of nickel foil is inserted between contact surfaces in order to improve the contact resistance at these locations.
(0.005") nickel foil band with a width of about 3.175-mm (0.125") is wrapped around the perimeter of the two interfaces of the sample with the meter bars on either end with a 0.254-mm (0.01") nichrome wire wrapped around the foil to hold it all in place as seen in Figure 4-4. The purpose of this is to assure alignment of the sample and meter bars during assembly. Figure 4-5 shows the finished assembly of the sample column.

The nickel guard is then screwed into place and diatomaceous earth powder is carefully filled in between the sample column and the guard as insulation. The remaining components of the assembly are put in place along with the spring to ensure a uniform constant contact pressure through the sample column. The assembly is laid horizontally where thermocouples are bonded with the Omega CC High Temperature Cement in small holes in the guard and a small sheet of high-temperature insulation is wrapped around the guard, as seen in Figure 4-6.

Once the assembly was completed with heaters in place, the measurement section is inserted into the tube furnace (Figure 4-7). It should be noted that it is placed in the same axial location which is photographically recorded each time a new sample is loaded. In this way, the effect of the furnace and the radiative heat sink should roughly be the same between installations.

![Figure 4-4. Photograph showing the top face of the cold-side meter bar before placement of sample with nickel foil and band in place.](image)
Figure 4-5. Photograph showing the assembly of test sample and meter bars with thermocouples cemented into place on the meter bars and test sample (middle).

All connections are made for routing thermocouple and power wires through the gas tight seals. At this point the furnace controller is programmed and set to run while the air inside the quartz tube is purged with the vacuum pump and backfilled with helium. This process is repeated several times to help ensure an inert He atmosphere for the testing being performed. The helium is then maintained at ~5-7.5-cm (~2–3") of water pressure throughout the tests.
4.2.2. Running Procedure

With assembly complete, the system is set to run at a certain temperature until steady-state conditions are met. Steady-state conditions for this experiment were defined such that the thermocouples’ readings vary no more than ±0.05 K/hr as recommended by ASTM E1225 [38]. Data was collected at a sampling rate of 1 sample per ten seconds (or 0.1 samples per second). Steady state was achieved when the data from each thermocouple for a number of points >360 (1 hour) had a standard deviation less than 0.025K (assuming a normal distribution, this equates to 95% of the data within ±2 standard deviations or 0.05 K of the setpoint temperature).
ASTM E1225 [38] recommends the guard temperature profile be matched to the sample column or set to the mean value for measurements of this type. Because of the design configuration used, which in general simplifies the design, the guard temperature configuration was set to be a combination of the two recommended methods. The hot end of the guard was set so that it would be slightly less than the point at the same cross-section in the sample column. In this way the temperature at the heat-sink end of the guard would be slightly more than the temperature at the sample column interface with the heat sink. In this way, the heat lost from the sample column to the guard in the hotter portion of the apparatus will be approximately equal to the heat gained in the colder portion. An example of a temperature versus distance plot from one of the measurements performed is shown in Figure 4-8 demonstrating this idea.

Later, finite element analysis has recently revealed that the ideal temperature profiles are so that the gradient of the guard temperature profile matches the temperature gradient of the sample itself. This result is discussed in detail in Section Error! Reference source not found.. All results to this point are using the previously described conditions shown in Figure 4-8.

![Figure 4-8. An example of typical temperature profile plots for the sample column and guard. The plot shows the approximate equality in temperature differences between the hot and cold portions of the sample column and the guard; heat lost in the hot end is approximately equal to heat gained in the cold end.](image-url)
4.2.3.  *Calculation*

Once steady-state conditions have been achieved, calculation of the thermal conductivity may be performed. The temperatures are averaged over the data points deemed to be steady-state (discussed in Section 4.2.2) and used in the calculation. The heat flow in the sample is found by averaging the heat flows in the meter bars, which may be found using Fourier’s Law for each bar as in Equation (3.5). The thermal conductivity at its average temperature may then be calculated as in Equation (3.6).
CHAPTER 5
UNCERTAINTY ANALYSIS

Two studies were performed to quantify and better understand the error associated with the measurement system design. The first was to perform a propagation of error analysis of Equations (3.5) and (3.6) demonstrating the precision error in each contributor of the measurement. To better understand the measurement system limits and capabilities as well as to better define the error associated with the system operating conditions, a simple yet effective finite element computational model was developed in the second study. Through the second study, a significantly better understanding and guidelines have been provided for designing systems based on the guarded-longitudinal-heat flow method.

5.1. Determinate Error Analysis

A detailed discussion of errors associated with this type of measurement is discussed in Reference [12] and in Reference [50]. As an initial analysis of the uncertainty associated with this experimental measurement system, a propagation of error analysis was performed on what are referred to as determinate errors in a manner similar to that performed by Sweet et al [50]. Determinate errors are simply the errors that can be estimated relatively accurately. At this stage, this analysis is not seen necessarily as the uncertainty in the final results but more as a guide to locating and reducing error contributions as calibration and testing continues.

Using the equations for sample thermal conductivity shown in Equations (3.5) and (3.6) and assuming that all of the independent variables which make up \( k \), are uncorrelated with the exception of the reference sample thermal conductivities in Equation (3.5) that come from the same source and have the same associated uncertainty, the variance of \( k \), may be found in Equation (5.1) where the relative variance is defined in Equation (5.2).
The uncertainty in the temperature differences, \( \delta \Delta T_m \) and \( \delta \Delta T_s \), were taken as given in Reference [50] as 0.15°C, which is much less than the 2.2°C or 0.75%, whichever is greater, recommended for Type N thermocouples. The reasons for the assumed better accuracy include: (1) modest temperature differences being measured, (2) thermocouple wire taken from the same spool, and (3) the same reference junction connections and readout devices [50].

An additional uncertainty arises in the uniform axial heat flow assumption. Sweet et al. showed that the error associated with non-uniformity is <1% if the stack thermal resistance is low compared to that of the surrounding insulation layer [50]. Therefore, it is included in this analysis as 1%. The uncertainties associated with each relative parameter are given in Table 5-1.

The result of the calculation shows that the contribution of determinate errors excluding the reference sample thermal conductivity uncertainty may be quite low, about 2–3%. By far the largest contributor to the error is found in the reference material thermal conductivity, which at best is about 5% by itself.

As was mentioned before, these results represent a preliminary analysis including determinate errors only. More attention will be given to precision at a later date as more samples are tested and the repeatability and reproducibility of the system are better known.
Table 5-1. Determinate uncertainties in measured parameters for $k_s$ showing contribution to overall uncertainty.

<table>
<thead>
<tr>
<th>Contributor</th>
<th>Variance, $\left{ \frac{\Delta x_i}{x_i} = \sigma_{x_i} \right} (x10^2)$</th>
<th>$\sigma_{x_i}^2(x10^4)$</th>
<th>Actual Contribution to Eq. (3) $(x10^4)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$k_m$</td>
<td>5</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>$A_m$</td>
<td>0.07</td>
<td>0.0049</td>
<td>0.00245</td>
</tr>
<tr>
<td>$\Delta T_m$</td>
<td>1.5</td>
<td>2.25</td>
<td>1.125</td>
</tr>
<tr>
<td>$\Delta Z_m$</td>
<td>0.34</td>
<td>0.1156</td>
<td>0.0578</td>
</tr>
<tr>
<td>$A_s$</td>
<td>0.07</td>
<td>0.0049</td>
<td>0.0049</td>
</tr>
<tr>
<td>$\Delta T_s$</td>
<td>1.5</td>
<td>2.25</td>
<td>2.25</td>
</tr>
<tr>
<td>$\Delta Z_s$</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Non-uniform flux</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Sum excluding $k_m$</td>
<td></td>
<td></td>
<td>5.44</td>
</tr>
<tr>
<td>Total Sum</td>
<td></td>
<td></td>
<td>30.44</td>
</tr>
<tr>
<td>Uncertainty excluding $k_m$</td>
<td></td>
<td></td>
<td>2.3%</td>
</tr>
<tr>
<td>Overall Uncertainty in $k_s$</td>
<td></td>
<td></td>
<td>5.5%</td>
</tr>
</tbody>
</table>

5.2. Numerical Uncertainty and Optimization Analysis

The objective of this section is to present the results of a numerical simulation of the comparative-guarded-axial heat flow measurement process in order to better understand and control the uncertainty associated with this technique in general, as well as to aid in obtaining an accurate thermal conductivity measurement for the TRISO fuel compact. This analysis provides guidelines to consider for the design of similar apparatus. The commercial software package, COMSOL Multiphysics was used in this steady-state heat transfer analysis.

5.2.1. Computational Setup

Figure 5-1 presents a schematic illustration of the cut-bar technique. Due to its axisymmetric geometry, the problem can be solved using a cylindrical coordinate system; thus a
three dimensional (3D) problem is reduced to two dimensions (2D). A specimen with an unknown thermal conductivity is sandwiched between a pair of meter bars with known thermal conductivity. A temperature gradient along the test stack (or sample column referring to the meter bars and test specimen) is created by keeping the hot end at \( T_{hb} \) and the cold end at \( T_{cb} \) so that the temperature difference \( \Delta T_{hc} \) between the two ends is a constant value. The sample column is surrounded by an insulation material. As was discussed in Section 4.1.2, diatomaceous earth powder was selected for use in the TFTCMS due to its low thermal conductivity [38] and
availability. This powder layer is encased by a rigid, thermal guard which has a linear temperature distribution from the hot end temperature, \( T_{gh} \), to the cold end temperature, \( T_{gc} \).

Based on the specimen thermal conductivity range, as well as the guidelines from the ASTM standard [38] and work performed by Didion [54], the materials of the meter bars and guard were chosen as stainless steel 304 and nickel alloy 201, respectively. The thermal conductivity of stainless steel 304, which increases nearly linearly with temperature, can be found in [57]. The thermal conductivity of nickel can be found in [59]. Both temperature dependent properties were input into COMSOL and interpolated with temperature.

The sizing of the geometries of the test system was designed according to the guidelines mentioned above and is presented in Table 5-2. To test the appropriate working range and minimize the overall error, parametric studies were performed on several parameters including: the length ratio of \( L_s/L_m \), aspect ratio of \( L_s/R_s \), mismatch ratio of \( R_s/R_m \), insulation \( k_i \) and its thickness ratio of \( R_i/R_s \), interfacial thermal resistance, guard-bar temperature mismatch \( \Delta T_{bg} \) and the average temperature deviation \( \Delta T_a \).

In the ASTM standard, two guarding temperature schemes are recommended: (1) guard temperature gradient matching the test stack temperature gradient and (2) isothermal guard with a temperature equal to the average temperature of the specimen. In this analysis, a parametric variation of the temperature difference between the guard and meter bars is carried out while keeping the two average temperatures identical. Thus if the guard hot end is \( \Delta T_{bg} \) °C cooler than

<table>
<thead>
<tr>
<th>( R_m )</th>
<th>( R_s )</th>
<th>( R_i )</th>
<th>( R_g )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00615</td>
<td>0.00615</td>
<td>0.022225</td>
<td>0.028575</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>( L_m )</th>
<th>( L_s )</th>
<th>( T_{cb} )</th>
<th>( T_{hb} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0254</td>
<td>0.0254</td>
<td>848.15</td>
<td>898.15</td>
</tr>
</tbody>
</table>
the hot end of the meter bar, the guard cold end is $\Delta T_{bg} {^\circ}C$ hotter than the cold end of meter bar. When $\Delta T_{bg}$ changes from zero to half of the temperature difference of the whole setup, $\Delta T_{hc}$, the guard temperature distribution varies from scheme (1) to (2). To separately quantify the effects of the guard temperature gradient and the average guard temperature, an additional temperature increase is applied to the guard for some cases, shifting its overall temperature gradient by an amount $\Delta T_a$. The boundary condition for the insulation is set according to the solution for steady-state, 1-D heat flow in the radial direction between two constant temperature surfaces.

$$T_i(r) = T_{bg, cg} + \Delta T_{bg} \frac{\ln(r/R_i)}{\ln(R_m/R_i)} \tag{5.3}$$

If the guard temperature matches the bar temperature, this reduces to a constant temperature applied on the boundary. On the outer surface of the guard, a linearized temperature distribution varying from $T_{gc}$ to $T_{gh}$ is applied. On the two axial end surfaces of both the guard and meter bars, constant temperatures are imposed due to the relatively large thermal conductivity of the meter bars and guard as compared with the insulation.

Inside the domain, the overall temperature distribution is calculated from the steady-state heat conduction equation,

$$\frac{1}{r} \frac{\partial}{\partial r} \left( k \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left( k \frac{\partial T}{\partial z} \right) = 0 \tag{5.4}$$

where the thermal conductivities of the various regions in the domain are defined in the nomenclature and are substituted into Equation (5.4) according to their corresponding location.

Thermal resistance may exist at the interfaces between the meter bars and the specimen due to imperfect contact. During model generation, a “pair” was created on the interface to link the meter bar and specimen domains. If thermal resistance is not present, a default continuity condition is applied on the interface where the temperatures and fluxes across the interfaces are equal. However, when thermal resistance is considered, a thin, thermally resistive layer is turned
on causing a temperature 'jump' across the interface while the flux across the interface is still equal. Mathematically, the boundary condition can be expressed as

\[ -k_s \left. \frac{\partial T_s}{\partial z} \right|_{z=L_m} = \frac{T_s - T_m}{R_{th}} = -k_s \left. \frac{\partial T_m}{\partial z} \right|_{z=L_m} \]  \tag{5.5}

Two methods were used in calculating the thermal conductivity of the specimen in the simulation. The calculated thermal conductivities for the specimen from both methods were compared to the true input values of the specimen. The first method has the purpose of mimicking thermocouple point measurements and locations as in the TRISO compact measurement apparatus. Using the fore described boundary conditions and input thermal conductivities for the simulation domain, the temperatures \(T_1\) thru \(T_6\) at positions \(Z_1\) thru \(Z_6\) can be monitored after reaching steady state. The calculation of thermal conductivity of the specimen can be performed based on the following equations adapted from the ASTM standard:

\[ q_1 = k_m \left[ \frac{(T_q + T_2)}{2} \right] \frac{T_2 - T_1}{Z_2 - Z_1} A_m \] \tag{5.6}

\[ q_2 = k_m \left[ \frac{(T_5 + T_6)}{2} \right] \frac{T_6 - T_5}{Z_6 - Z_5} A_m \] \tag{5.7}

\[ k_{sc} = \frac{1}{2} \frac{q_1 + q_2}{A_s} \frac{Z_4 - Z_3}{T_4 - T_3} A_m \] \tag{5.8}

In the simulation, a numerical integration of temperature and heat flux over the interface or any arbitrary surface is possible. Thus, a second, more accurate scheme for obtaining specimen thermal conductivity uses integration. The values of \(q_1\) and \(q_2\) are obtained by averaging the heat flow through both ends of the hot and cold side meter bars, respectively. In the same manner, \(T_3\) and \(T_4\) in Equation (5.8) are found by integrating over the two ends of the test specimen. The distance between \(Z_3\) and \(Z_4\) becomes the specimen length, \(L_s\).
5.2.2. Preliminary Tests

Two-dimensional structured grids were used in the simulations. To ensure reliable results, grid independence was tested using four mesh sizes. The first case used 30 divisions in the axial (z) direction and 25 divisions in the radial (r) direction. The mesh size of the three consecutive cases was increased by a multiple of 2 in each direction in relation to the previous one. Using the densest case as a reference, the relative deviation from the reference of the resulting $k_\text{sc}$ obtained from Equation (5.8) for each mesh density, is plotted in Figure 5-2. For a large range of $k_\text{s}$ (20<$k_\text{s}$<100), even the coarsest mesh has an error of less than 0.002%. At the lowest studied $k_\text{s}$, the maximum error is still less than 0.06%. For other denser cases, the deviations are all less than 0.002% over a specimen $k_\text{s}$ range of 5-100 W/m/K. Because the resulting deviations are small, the error induced by the tested mesh densities is assumed negligible.

Figure 5-3 presents a comparison of the calculated thermal conductivity between: (1) using point temperature monitors $T_1$-$T_6$ at the specified positions $Z_1$-$Z_6$ and (2) employing integration of temperature and heat flux over the previously specified surfaces. Unless additional

![Figure 5-2. Grid independence test for different specimen thermal conductivity input.](image-url)
Thermal conductivity of sample (W/m/K)

Percentage error (%)

Calculation by point temperature monitors
Calculation by heat flux monitors

Explanation is given, all parameters are set to those listed in Table 1 and $\Delta T_{bg}=\Delta T_{a}=0$ which means that the guard has a linear temperature gradient matched at the hot and cold ends to the corresponding axial locations (the ends) of the sample column. The calculation of $k_{sc}$ in Equation (5.8) relies on an assumption that equal heat flow occurs through the cross sections of both meter bars and the specimen. If it is true, computation of $k_{sc}$ by Equation (5.8) should not incur too much systematic error. In reality, however, the constant heat flow assumption is rather weak due to the radial heat exchange with the guard and axial heat flow shunting at the specimen and meter bar interfaces due to their different thermal conductivities. These results can be seen from the percentage error plot in Figure 5-3 for varying specimen thermal conductivity.

If $k_s=k_m$, the sample column virtually becomes one bar from the perspective of the heat flow. Since no temperature gradient is observed from the bar to the guard, there is no heat flow in the radial direction. The measurement does not incur any error due to a perfect 1D (axial) heat flow condition. As $k_s$ deviates increasingly from $k_m$ ($k_s \neq k_m$), the induced error becomes increasingly larger. Meanwhile, the lower $k_s$ case ($k_s << k_m$) generates larger error compared with higher $k_s$. The reason is that, in addition to axial heat flow shunting, radial heat exchange

Figure 5-3. Difference of percentage error of calculation by heat flux integration and point temperature monitors.
becomes more significant as \( k_s \) becomes smaller (closer to \( k_i \)). For \( k_s = 5 \text{ W/m/K} \), the ratio of \( k_s/k_i \) is roughly 50. For this condition, the error is still only around 11%. If this ratio becomes larger so that the radial heat flow is obstructed, the calculated error becomes smaller. Another important observation is that the error resulting from low \( k_s \) (\( k_s < k_m \)) is positive (calculated value is larger than input one) whereas the error brought about by high \( k_s \) (\( k_s > k_m \)) is negative since the measurement results always tend toward \( k_m \).

A comparison of the two curves indicates that the error from heat flux integration is always smaller, as would be expected. The point measurements on the bar surfaces are affected more by the radial temperature gradient while averaging through integration reduces the effect of the radial temperature gradient. Thus calculation by integration over the surface is more accurate. Because in the TRISO compact experimental measurement system the thermocouples must be attached to the sample surface due to the requirement of non-destructive measurement, the following analysis employs Equation (5.8) using surface, point measurements for thermal conductivity evaluation.

5.2.3. Numerical Results and Discussion

Figure 5-4 presents the effect of the thermal conductivity of the insulation material on the error in the measurement. Instead of using the value of thermal conductivity of diatomaceous earth powder, \( k_i \) was changed parametrically from 0.001 W/m/K to 0.2 W/m/K. If \( k_i \) is very low, in the range of ~0.001 W/m/K, the setup renders very accurate results without appreciable system error for all \( k_s \) considered. Under this condition, axial heat flow shunting is reduced even for significantly differing specimen and reference bar thermal conductivities. Additionally, radial heat exchange is blocked due to small \( k_i \) (\( q'' \propto k_i \)). For values of \( k_s \) close to \( k_m \) the effect of the insulation is nearly negligible. As \( k_s \) increasingly differs from \( k_m \), the effect of axial heat flow shunting and radial heat flow becomes more significant, thus, rendering more error.
Figure 5-4. Percentage error variation as thermal conductivity of insulation material varies parametrically.

Because an “ideal” insulator is not available (especially for use over the required temperature range), an alternative way to mitigate radial heat flow is to increase the insulation thickness. Figure 5-5 presents the deviation of calculated \( k_{sc} \) for different ratios of \( R_i/R_s \) from the \( k_{sc} \) obtained with a very thick insulation layer (\( R_i/R_s=11.46 \)). The value of \( k_{sc} \) for \( R_i/R_s=11.46 \) was

Figure 5-5. Deviation of results with different insulation layer thickness from specimen thermal conductivity with very large insulation thickness.
selected as the reference, instead of input $k_s$, so that the effect of insulation thickness is emphasized. For any $k_s$, the deviation approaches a limiting result as the ratios of $R_i/R_s$ increases. Accordingly, as $R_i/R_s$ becomes larger than the critical value, roughly 5 in this case, the influence of additional thickness becomes negligible. It should be noted that some system error still exists under this condition due to axial heat flow shunting.

Figure 5-6 presents the effect of the sample column length on the resulting percentage error. Note that the ratio of $L_s/L$ increases with a decrease of $L_m$ since $L=2L_m+L_s$. From the point of view of systematic error, reducing the length of the meter bars is helpful to reduce error. As the meter bar length approaches zero, the sample column tends toward the case of a single, solid specimen bar meaning that the radial heat flow and axial heat flow shunting tend toward zero. From the propagation of error analysis (see Equation (5.1)), however, reducing meter bar length increases the uncertainty since $x_i$ in Equation (5.2) tends to zero. Both types of error must be considered comprehensively during design.

Figure 5-6. Percentage error variation when meter bar length changes parametrically.
When aspect ratio \( L_s/R_s \) is changed parametrically by varying the length of specimen \( L_s \) or radius \( R_s \), the resulting error is presented in Figure 5-7 displaying a trend similar to that in Figure 5-6. For these calculations the length ratio, \( L_s/L \), and the insulation thickness ratio, \( R_s/R_i \), are maintained at 0.333 and 3.61, respectively. Thus the effects of these two parameters are eliminated. With a reduction of specimen length or with an increase of specimen radius, the specimen geometry changes from a cylinder/bar to a plate/disk. The reason for the decrease in system error with a flatter specimen resides in the fact that a reduction of specimen length or an enlargement of radius enhances the ratio of axial heat flow to radial heat flow because the ratio of axial conducting area to circumferential surface (radial conducting) area becomes larger. Reasoning in a manner similar to as was done for Figure 5-6, the precision error thereby increases if specimen length is reduced (see Equations (5.1) and (5.2)). However, augmenting the sample diameter improves the system accuracy without affecting the measurement precision error. Thus the sample diameter should be as large as possible with consideration of heaters and furnace size.

Figure 5-7. Percentage error variation when specimen length to radius ratio \( (L_s/R_s) \) changes parametrically.
Figure 5-8 presents the response error when specimen and meter bars have differing radii. If \( R_s < R_m \), the calculated specimen thermal conductivity is always larger than for when \( R_s = R_m \). Since positive error occurs for low \( k_s \) and negative error results for high \( k_s \) when \( R_s = R_m \), low \( k_s \) cases render more error than do high \( k_s \) cases when \( R_s < R_m \). The opposite trend is true for the cases when \( R_s > R_m \). When \( R_s < R_m \) the axial heat flow in the specimen is lower than that in the meter bars. Therefore, when the equal heat flow assumption is employed in Equation (5.8), the calculated \( k_{sc} \) is higher than that obtained when \( R_s = R_m \). A similar reasoning applies to the cases when \( R_s > R_m \) and their calculated \( k_{sc} \) is lower than that obtained when \( R_s = R_m \). The simulation results are consistent with the conclusion drawn by Babelot [52] in their experiment where an overestimation of thermal conductivity values was observed when the sample diameter was reduced from the diameter of their meter bars, 10-mm, to 5-mm.

Thermal resistance at the interfaces of the specimen and meter bars cannot be fully avoided due to imperfect contact; thus it is necessary to quantify its effect. Figure 5-9 presents a comparison of results for varying contact resistance to that of no resistance for different specimen thermal conductivities. When thermal resistance is relatively small (< 1e-5 m²K/W) the influence

![Figure 5-8](image-url)

**Figure 5-8.** Percentage error with respect to specimen \( k_s \) when specimen radius changes, \( R_s \neq R_m \).
Thermal resistance on specimen and meter bar interfaces ($m^2K/W$)

Deviation from no resistance situation (%)

-1.5 -1.0 -0.5 0.0 0.5 1.0

$k_s=5$ W/m/K
$k_s=15$ W/m/K
$k_s=25$ W/m/K
$k_s=50$ W/m/K
$k_s=100$ W/m/K

Based on no resistance

Figure 5-9. Deviation of results with $R_{th}$ from the calculated thermal conductivity without $R_{th}$ when thermal contact resistance ($R_{th}$) increases parametrically.

of thermal resistance is insignificant. With a further increase of $R_{th}$, the calculated $k_{sc}$ deviates increasingly from the value corresponding to no contact resistance. Axial heat flow shunting is enhanced by the block of axial heat flow by the resistances at the interfaces. Using the temperature gradients in the meter bars and specimen as well as the temperature measurements at locations $Z_2$ to $Z_3$ and $Z_4$ to $Z_5$ to extrapolate temperatures to the interfaces, the magnitude of the interfacial thermal resistances may be calculated in the actual measurement system.

Figure 5-10 presents the percentage error generated by a comparison of computed thermal conductivity ($k_{sc}$) to the input true value ($k_s$) when $\Delta T_{bg}$ is varied parametrically. Over the working temperature range, the thermal conductivity of stainless steel 304 is approximately 24 W/m/K. When $\Delta T_{bg}=0$, the error is negligible for a specimen with $k_s=25$ W/m/K because the radial temperature gradient between the meter bar and the specimen is not significant. However, for an increasing divergence of $k_s$ from $k_m$, the heat exchange and shunting in the radial and axial directions have more impact on error generation. Similar to Figure 5-4, low specimen $k_s$ is affected more than the high $k_s$ cases due to its low ratio of $k_r/k_i$. 
Figure 5-10. Percentage error variation with respect to the change of bar and guard temperature difference $\Delta T_{bg}$.

When $\Delta T_{bg}$ increases from zero to half of $\Delta T_{hc}$, the corresponding errors from all of the differing $k_s$ cases change linearly with positive slope. For $k_s<k_m$, the errors increase in magnitude continuously. For $k_s\approx k_m$, the error increases from roughly zero at the "matching" condition to around 6% at the "isothermal" condition. However, for $k_s>k_m$, the negative errors approach zero (the critical value of $\Delta T_{bg}$) with an increase of $\Delta T_{bg}$ and continue to increase becoming positive after passing the critical value. Based on this analysis, it seems that the two recommended guard working conditions are not optimized and need to be considered more fully. If $k_s>k_m$, a lower temperature gradient on the guard compared to the sample stack is helpful for eliminating systematic error. Thus, the optimum $\Delta T_{bg}$ is around 8 °C for $k_s=50$ W/m/K and 13 °C for $k_s=100$ W/m/K.

Since a lower temperature gradient on the guard is better for large $k_s$, it is reasonable to assume that, in order to reduce error, a higher temperature gradient should be used on the guard for small $k_s$. For $k_s=15$ or 5 W/m/K cases, one can observe that the errors approach zero with a negative increase of $\Delta T_{bg}$, viz. an increase of temperature gradient of the guard. Beyond a critical
$\Delta T_{bg}$, the error becomes negative and increases in magnitude with a further increase of negative $\Delta T_{bg}$. Thus for $k_s=15$ and 5 W/m/K, the critical $\Delta T_{bg}$ is around -6 °C and -21 °C, respectively.

Figure 5-10 indicates that the temperature gradient on the guard significantly affects the accuracy of the calculated specimen $k_{sc}$ using Equation (5.8). Figure 5-11 presents the temperature distributions along the sample column and guard for critical values of $\Delta T_{bg}$ for various $k_s$. When the guard temperature matches the test stack temperature, both the hot end and cold end surfaces as seen in Figure 5-1 have isothermal boundary conditions. This situation is equivalent to a condition where the stack, insulation, and guard domains are wholly covered by a large isothermal heat source and heat sink. For the $k_s=5$ W/m/K case, the temperature gradient is larger in the specimen than that in the meter bars. Since the hot-side (cold-side) meter bar has a higher (lower) temperature than the corresponding height on the guard portion, radial heat flows from the hot-side meter bar to the guard (guard to cold-side meter bar). The amount of heat transferred radially, however, is affected by the amount of axial heat flow shunting as well.

The difference of temperature distributions in the test stack and guard creates a radial temperature

![Graph](image)

**Figure 5-11.** Temperature distributions along test stack and guard surface demonstrating ideal temperature distributions for various sample thermal conductivities.
gradient inducing radial heat flow and is the main reason for the error. For the $k_s=100 \text{ W/m/K}$ case, the heat flow direction is reversed between the hot and cold ends of the sample stack.

The guard temperature gradient corresponding to the critical value of $\Delta T_{bg}$ (the systematic error tends toward zero as shown in Figure 5-10) is superimposed in Figure 5-11. Under such circumstances, the guard temperature gradient tends to vary from "whole stack match" toward "specimen gradient match" but still having a slight deviation from this condition. The change of guard temperature gradient induces a slight test stack temperature distribution change, but this variation is too small to be shown. The little re-distribution of temperature minimizes the systematic error. According to Figure 5-10 and Figure 5-11, the optimum $\Delta T_{bg}$ is primarily influenced by the specimen thermal conductivity.

In the real TRISO experimental measurement setup, the temperature gradient of the guard is easily manipulated as desired but achieving equal average temperatures of both the guard and the specimen is difficult since the temperature distribution of the specimen is affected both axially and radially. Figure 5-12 presents the deviation of results when guard and specimen average temperatures have a difference of $\Delta T_a$. The deviation is calculated based on the case

![Figure 5-12. Percentage deviation with respect to the change of $\Delta T_a$ from the calculated thermal conductivity when $\Delta T_a=0$.](image-url)
when $\Delta T_a=0$. For generic consideration, $\Delta T_{bg}$ is set to 10 °C. From the figure one can see that the deviations are relatively small for all $k_s$ cases (<0.05%). Thus one can conclude that once an appropriate temperature gradient is imposed on the guard, the average temperature difference between the specimen and the guard does not affect the accuracy significantly. Comparing Figure 5-12 and Figure 5-10, the relative importance of the guard temperature gradient and the average guard temperature is clearly apparent. These conclusions are convenient and useful for simplifying the design of such apparatus as well as their working conditions.

5.2.4. **Modeling Conclusions**

The uncertainty associated with the guarded-comparative-longitudinal heat flow technique was analyzed from the systematic and precision error points of view. It was found that if the sample diameter is restricted, a compromise has to be made geometrically considering both types of error. Low meter bar to specimen length ratio and low specimen aspect ratio are better for reducing systematic error. Large distance between temperature monitor positions, however, is helpful in reducing precision error. If the specimen diameter can be increased for a given length, the bias error will be improved while not affecting the precision error. Particularly, having a large aspect ratio is more beneficial for low thermal conductivity specimens.

Large interfacial thermal resistance increases the system error. It blocks the axial heat flow thus the effect of axial heat flow shunting is more significant. A conducting medium at the interfaces should be used to reduce thermal resistance such as highly thermally conductive grease, or for higher temperatures, a thin metal foil.

The temperature gradient on the guard plays an important role in reducing the system error without deteriorating the precision error. The ideal guard temperature condition to reduce system error is achieved by matching its temperature gradient to the sample temperature gradient, not necessarily by matching the guard temperature to the test stack at the hot and cold ends. An
evaluation of the deviation of guard average temperature from the specimen average temperature indicates that it has an insignificant effect on the measurement accuracy.
CHAPTER 6
EXPERIMENTAL RESULTS AND DISCUSSION

The results and discussion from several validation samples and two NGNP graphite samples are presented in this section.

6.1. Validation Samples

Because the thermal conductivity of the graphite fuel material is not yet known, several samples were selected to validate the system’s performance covering the range of possible expected values. Table 6-1 shows a list of the samples selected to accomplish this testing.

The samples were selected to cover a range of expected possible thermal conductivity values. Inconel 625, SS 304, and high purity iron have thermal conductivities in the lower, middle, and upper portions of the expected range, respectively. Thermal conductivity data for each of these materials is available in literature as a source of comparison.

It is worth noting that a glass-ceramic thermal conductivity reference material is also being considered for testing the low thermal conductivity range and to provide an even better validation of the system performance as it is a standard reference material which may be acquired.

Table 6-1. Materials selected for apparatus calibration and validation.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal Conductivity, W/m/K (For T&lt;sub&gt;s&lt;/sub&gt; = 100°C-900°C)</th>
<th>Reason for testing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inconel 625</td>
<td>10–20 [61-62]</td>
<td>Has a range slightly lower than the expected values for the fuel compacts, data available for comparison</td>
</tr>
<tr>
<td>Stainless steel 304</td>
<td>14–30 [57-59]</td>
<td>Well-defined thermal conductivity in literature, in expected range of the fuel compacts</td>
</tr>
<tr>
<td>High purity Fe (99.95%)</td>
<td>70-30 [59]</td>
<td>Thermal conductivity in the upper expected range of the fuel compact, data available in literature, provide information about higher conductivity capabilities</td>
</tr>
</tbody>
</table>
from the Institute for Reference Materials and Measurements (IRMM). This glass-ceramic material has a thermal conductivity range of ~4 W/m/K to ~2.5 W/m/K between 100°C and 700°C.

6.1.1. Measurement of Stainless Steel 304

The first overall sample measured in the experimental system was stainless steel 304, the same material as the meter bars. The sample was machined to the approximate size of a fuel compact with a length of 25-mm and a diameter of 12.3-mm. The sample was first measured at 300°C under various environmental settings. Measurements were taken to see the effects under the following settings: sample heater and guard heaters both on, sample heater on with guard heater off, and no heaters on. Measurements were also performed with an air atmosphere as well as with an inert, helium atmosphere to see temperature profiles under these conditions. From these tests, measurement design conditions verified that the ideal condition is to have a helium atmosphere with both the guard and sample heaters turned on, setting the guard temperature to provide for conditions discussed in the previous section. The experimental section was then removed from the furnace, disassembled, and examined.

The same sample was again assembled in the system, and measurements were performed from about ~200°C up to ~600°C at increments of 50°C using a helium atmosphere. The results were compared to the data from Bogaard [57] recommended by Sweet [50] in a report on comparative thermal conductivity measurement methods as well the results of Graves et al [58]. The results are plotted in Figure 6-1.

The data used for the reference sample thermal conductivity is that of Bogaard as it is recommended by Sweet [50] and has a given uncertainty of ±5%. The results vary no more than 3% from Bogaard for temperatures between 300°C and 600°C. For this testing, data was not collected above 600°C because the 0.076-mm (0.003”) Type N thermocouple is not
Figure 6-1. Measured thermal conductivity of stainless steel 304L compared to recommended values from Bogaard [57].

recommended for long-term use above this temperature. Bare, fine gage, Type N thermocouple may experience significant decalibration with long-term use at high temperatures (see Section 4.1.2). For this reason, as mentioned previously, 0.127-mm (0.005”) size Type N thermocouple or larger or platinum-type thermocouples will be used for future measurements.

6.1.2. Measurement of 99.95% Pure Iron

In order to test higher thermal conductivity measurement ability, the second validation sample and third overall sample measured in the TFTCMS was 99.95% pure iron from ESPI Metals. The sample was cut to the approximate length of a fuel compact and the end surfaces were polished. The dimensions of the iron sample were measured to be a length of ~25.648mm and a diameter of ~12.813mm (slightly larger diameter than the meter bars).

Due to the decalibration of the smaller thermocouples, 0.127mm (0.005”) Type N thermocouple was used on the iron sample. Decalibration of the thermocouple is still an expected problem at high temperatures, but the larger thermocouple size is more resistant to
contamination, much easier to handle, and still small enough to not contribute much to the overall uncertainty. A photograph of the iron sample with thermocouples in place is shown in Figure 6-2.

Measurements were performed from 100°C to 600°C at increments of 50°C. Each temperature was then measured a second time in reverse order. Measurement temperatures were then run between 600°C and 800°C, again each temperature was measured twice. Figure 6-3 displays the measured thermal conductivity of 99.95% pure iron compared to the values recommended by the TPRC data series [59] for 99.99% pure iron.

The results show good agreement with the published values for 99.99% pure iron. For 100°C to 600°C, the difference is < 8% for all temperatures. As can be seen in the figure, for > 600°C the deviation becomes greater, between 5 and 10% for the first set of points collected in this range and between 12 and 14% for the second set. The results seem very promising even for a sample with a higher conductance than the meter bars. For temperatures above 600°C, the deviation becomes larger but the overall trend of the curve is consistent.

Figure 6-2. 99.95% pure Fe sample prior to thermal conductivity measurement.
6.1.3. Measurement of Inconel 625

A sample of inconel 625 was obtained from ESPI metals and was cut to a length of 25.62mm with a diameter of 12.646mm. 0.127mm (0.005”) Type N thermocouples were used for all temperature measurements used in calculating the thermal conductivity of the sample. Measurement order and temperatures was similar to that of the pure iron sample as discussed in the previous section except the maximum temperature was run up to 900°C. Figure 6-4 shows a photograph of the inconel 625 sample before measurement.

The results are shown in Figure 6-5 compared to published values. The results compare very well to the data given by www.hightempmetals.com [61]. The maximum difference is 5.8%, near 600°C, over the range of available data. The data from the Battelle Memorial Institute [62] shows a larger difference over the entire temperature range.
Figure 6-4. Photograph of inconel 625 sample to be measured by the TFTCMS.

Figure 6-5. Measured thermal conductivity of inconel 625 compared to recommended values [61-62].

6.2. NGNP Graphite Samples

Two NGNP graphite samples were measured for the initial testing phase of the project in order to help validate the system’s capability to measure non-metallic and composite specimens.
The first sample was a composite, surrogate NGNP TRISO compact, and the second was a pure graphite sample.

6.2.1. *Measurement of NGNP Graphite/ZrO$_2$ Surrogate Compact*

A surrogate compact was provided by Oak Ridge National Laboratory (ORNL) made up of a graphite matrix with zirconium dioxide (ZrO$_2$) particles in place of the TRISO-coated particles. The particle volume fraction of the particles in the sample was approximately 30-38%. In order to test the potential capability to measure a sample of the form and similar composition of the TRISO compacts and to comply with SOW-7214, this surrogate compact was the second material measured in the experimental system. The sample’s dimensions were measured to be 25.5-mm in length with a 12.32-mm diameter. 0.076-mm (0.003”) Type N thermocouples were used for this sample. Figure 4-3 shows a photograph of the sample with thermocouples installed on the surface. Measurements were performed from 100°C up to 800°C at 50°C increments. Results are shown in Figure 6-6.

![Figure 6-6](image-url)

Figure 6-6. Values of thermal conductivity for graphite with ZrO$_2$ particles measured by the experimental system. Number next to data point indicates the order that data was collected. Set number represents order of data collection for a given temperature.
A very obvious and interesting observation about these results is the nearly constant value for thermal conductivity over a wide temperature range. A review of thermal conductivity values for amorphous graphite reveals it can have nearly any value and trend at a given temperature [63]. In addition, ZrO₂ reveals a slightly increasing value over the measurement temperature range [63]. Figure 6-6 shows the order in which data was collected with a numeric order label next to each data point, the order being random. Initially, two sets of data were collected at each temperature between 100 and 600°C (2 points/temperature). At that point, temperatures were then ramped above 600°C for the first time. The 0.076-mm (0.003”) Type N thermocouples used for these measurements are not recommended for use above about 600°C. For this reason, data was first collected below this point. The first set of data collected between 600 and 800°C showed a definite upward trend which, for now, is questionable.

The second set of data collected through this range shows a notable downward shift. This shift is also a point of interest and is believed to be due to possible decalibration of the thermocouples (the decalibration was a result of a combination of the high temperatures, small wire diameter, and thermocouple wire contamination by impurities). The third set of data collected, marked by a box, was to determine if the downward shift would carry over to lower temperatures, which was confirmed during testing as seen in Figure 6-6. The available options to correct this problem are to use either Type S thermocouples that have a much higher temperature usage range or a larger diameter Type N, which should be less susceptible to decalibration issues. After this measuring this sample all measurements were carried out using the larger, 0.127mm (0.005”) Type N thermocouples.

An unexpected observation made during post-measurement examination was that the graphite/ZrO₂ sample experienced a slight change in dimensions. The pre-test measured length and diameter was 25.5-mm and 12.32-mm, respectively. Post-test, they measured 25.35-mm and 12.35-mm. One possible explanation (though unlikely) for this change is the high temperature
and compressive force of the spring used in thermal conductivity testing. The spring used is rated at 18.5 lbs, giving a pressure of nearly 94 psi on the sample. The effect of this change in length on the calculated value of thermal conductivity is only about 1%. This phenomenon may be tested by using a spring of a lower spring constant.

6.2.1.1. Comparison with German Legacy Data

Figure 6-7 shows a comparison of the first set of data captured for each temperature between 100°C and 600°C and the data from Gontard [53]. Emphasis should be made that this is not a good direct comparison as the German data is for a different particle and much ambiguity surrounds the source of the data. The comparison should be taken very lightly. The legacy data shows a definite downward trend, while the NGNP graphite/ZrO$_2$ samples maintain an almost constant value. At lower temperatures there is about a 50% difference in thermal conductivities, while at higher temperatures the legacy data seems to approach the value of the ORNL sample.

Figure 6-7. Comparison of graphite/ZrO$_2$ sample measured by the TFTCMS to the legacy data for German TRISO fuel [53].
The lower thermal conductivity of the NGNP compact is attributed to it having graphite with much lower density than the German data.

6.2.2. Measurement of AGR-2 Graphite Matrix-Only Compact

The second NGNP graphite material tested in the TFTCMS was an AGR-2 graphite matrix only compact. The original size of this compact was unsuitable for use in the TFTCMS. The overall length of the sample was measured to be 16.8-mm and although shorter than the ideal case of 25-mm, was deemed acceptable. The outer diameter of the cylindrical compact was too large and was machined down to a diameter of 12.3-mm. All measurement thermocouples were fabricated from 0.127mm (0.005”) Type N thermocouple wire. Figure 6-8 shows the AGR-2 sample.

Figure 6-9 shows measured thermal conductivity values for the AGR-2 sample. Measurements were taken from 100-900°C following the order listed in the figure. The results show an interesting trend. As temperature is ramped up past ~450°C, thermal conductivity values increase. When temperature is ramped back down, thermal conductivity values remain fairly constant. The apparent permanent change of value is surprising.

Figure 6-8. Photograph of AGR-2 sample with thermocouples mounted awaiting application of high temperature cement.
The initial measured value of thermal conductivity of the AGR-2 sample was around ~10 W/m/K. After increasing up to 600°C, the value had increased to about ~13 W/m/K and there remained nearly constant over a complete cycling to 100°C and back. From 600°C to close to 800°C, the value increased to nearly ~16 W/m/K and again appeared to remain nearly constant at with changes of temperature, both up and down.

Post-measurement examination showed that the sample experienced shrinking (as did the graphite/ZrO$_2$ sample. The resulting shrinkage was measured to be approximately ~4.6% in length and ~1.4% in diameter (or overall volume shrinkage of ~7%). To date, the reason for this shrinkage is not clear. In fabrication the samples are pressed and sintered at 1700°C to 1800°C; this measurement does not come close to those temperatures. It is clear that the sample is fundamentally changing somehow, and therefore the thermal conductivity results are not too surprising. In order to help make a determination of the causes for these effects in the results, the same AGR-2 sample will be reassembled and measured to check to see if the value is close to last
measured values (~16 W/m/K). Future graphite measurements will also include a weight measurement of the sample before and after testing. Also more information about the sample history may provide additional clues as to why the sample shrunk and experienced a permanent change of thermal conductivity with high temperatures.
CHAPTER 7
CONTINUED WORK AND CONSIDERATIONS

Much work will continue in the area of sample measurement. Several samples are waiting to be measured by the TFTCMS. Additionally, work is now beginning and will continue in order to extend the temperature range of the measurement with the proper selection of materials and equipment as well as come up with a predictive model for thermal conductivity of TRISO fuel.

7.1. Ongoing Work

7.1.1. System Validation/Calibration

With the success demonstrated by the TFTCMS, an entire second identical system has been established to aid in collecting data of the many samples to be measured. With the two fully operational systems, measurements will continue to verify the finite element results and assure proper operating conditions for samples. A stainless steel 304 sample will be reassembled in the system in order to verify the finite element conclusions regarding guard temperature gradient and average temperatures as discussed in Section 5.2.4. The thermal conductivity of already measured samples will be verified using an alternate measurement method (most likely a laser flash, dilatometer, and differential scanning calorimeter; their combined results will give thermal conductivity as discussed in Section 3.2.1.2) as yet another comparison for validation of the results of the TFTCMS.

7.1.2. Surrogate/Graphite Testing

Several surrogate compacts and graphite samples have been provided by ORNL for measurement. Table 7-1 shows a summary of these materials and a prospective order for measurement based on information from Idaho National Laboratory (INL). ORNL and/or
Table 7-1. Table of surrogate compacts and graphite materials provided by ORNL.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Label</th>
<th>Size, inches (Diameter x Length)</th>
<th>Status/Order</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Graphite Only - INL</td>
<td>N/A</td>
<td>5x1</td>
<td>NM</td>
<td>Different graphite, less important than those from ORNL</td>
</tr>
<tr>
<td>Graphite Only - ORNL</td>
<td>AGR2-9</td>
<td>.6x.8</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>AGR2-10</td>
<td>.6x.8</td>
<td>M</td>
<td></td>
</tr>
<tr>
<td></td>
<td>AGR2-11</td>
<td>.6x.8</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Vector T1 - MO-15</td>
<td>.6x.8</td>
<td>NM - 3</td>
<td>Vector T1 and Vector T2 are made of a graphite only matrix of two different formations of natural graphite, synthetic graphite and resin</td>
</tr>
<tr>
<td></td>
<td>Vector T1 - MO-16</td>
<td>.6x.8</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Vector T1 - MO-17</td>
<td>.6x.8</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Vector T2 - MO-18</td>
<td>.6x.8</td>
<td>NM - 4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Vector T2 - MO-19</td>
<td>.6x.8</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Vector T2 - MO-20</td>
<td>.6x.8</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td>Graphite/ZrO2 particles - ORNL</td>
<td>N/A - Sample 1</td>
<td>5x1</td>
<td>M</td>
<td></td>
</tr>
<tr>
<td></td>
<td>N/A - Sample 2</td>
<td>5x1</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>N/A - Sample 3</td>
<td>5x1</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td>Graphite/TRISO HfO2 - ORNL</td>
<td>3UHF 9</td>
<td>.5x1</td>
<td>NM - 1</td>
<td>Contain 300 micron hafnia kernels and the standard coatings; nominally 100 micron thick buffer layer, 40 micron IPyC layer, 35 micron SiC and 40 micron OPyC</td>
</tr>
<tr>
<td></td>
<td>300HF 10</td>
<td>5x1</td>
<td>NM</td>
<td></td>
</tr>
<tr>
<td></td>
<td>500HF 9</td>
<td>5x1</td>
<td>NM - 2</td>
<td>Contain 500 micron hafnia kernels and the standard coatings</td>
</tr>
<tr>
<td></td>
<td>500HF 10</td>
<td>5x1</td>
<td>NM</td>
<td></td>
</tr>
</tbody>
</table>

Babcock and Wilcox may also provide additional surrogate samples that may be included in later testing using different graphite resins and/or packing fractions of surrogate TRISO particles.

7.2. Considerations for Other Operating Conditions

To further investigate higher temperature measurement, a 1600°C tube furnace will be setup and configured to measure samples to temperatures close to 1500°C. The conceptual design should not have to change significantly for any difference of working environment. Most of the difficulties involved with adaptation for different operating conditions are related to materials. In particular, materials appropriate for high temperature that can be protected from any corrosion or degradation must be selected.
7.2.1. Higher Temperature Measurement

The TFTCMS is currently capable of operation in a temperature range of 200 to 900°C and with slight modification could be pushed to 1000°C with a different sample column heater. The current heater has a limit of about 800°C (but has been successfully run up to 900°C). At temperatures higher than ~1000°C, the corrosion resistance of stainless steels becomes more of an issue, and alternate materials may need to be used in place of the 304 SS. A full investigation and selection of materials will be performed. Some material properties are listed in Table 7-2 and Table 7-3.

Thermocouple type will also become an issue. Type K and Type N thermocouples are able to withstand temperatures up to 1260°C but can experience high-temperature drift due to contamination and other causes. They may be used up to these high temperatures when protected by sheaths and at diameters that are significantly larger than is possible to use for the sample size of a TRISO compact. One option is to use platinum-type thermocouples (R or S) that have a much higher temperature limit of 1450°C and are not as susceptible to drift. The downside is that they are very costly.

Table 7-2. High-temperature wire properties [64].

<table>
<thead>
<tr>
<th>Material</th>
<th>Melting Temperature (°C)</th>
<th>Maximum Recommended Working Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molybdenum</td>
<td>2610</td>
<td>1900</td>
</tr>
<tr>
<td>Tungsten</td>
<td>3380</td>
<td>2200</td>
</tr>
<tr>
<td>Tungsten-5% Rhenium</td>
<td>3350</td>
<td>2300</td>
</tr>
<tr>
<td>Molybdenum-50% Rhenium</td>
<td>2550</td>
<td>2200</td>
</tr>
<tr>
<td>Niobium</td>
<td>2468</td>
<td>1800</td>
</tr>
<tr>
<td>Tungsten-26% Rhenium</td>
<td>3120</td>
<td>2300</td>
</tr>
<tr>
<td>Tantalum</td>
<td>2996</td>
<td>2400</td>
</tr>
<tr>
<td>Rhenium</td>
<td>3180</td>
<td>2400</td>
</tr>
<tr>
<td>Chromium</td>
<td>1907</td>
<td>1400(^\text{a})</td>
</tr>
<tr>
<td>Nickel</td>
<td>1453</td>
<td>1100(^\text{a})</td>
</tr>
</tbody>
</table>

a. estimated
Table 7-3. Material properties for sheathing materials used on high temperature probes [64].

<table>
<thead>
<tr>
<th>Material</th>
<th>Melting Temperature (°C)</th>
<th>Maximum Temperature ** in Air (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type 304</td>
<td>1454</td>
<td>927</td>
</tr>
<tr>
<td>304L</td>
<td>1454</td>
<td>927</td>
</tr>
<tr>
<td>310</td>
<td>1454</td>
<td>1149</td>
</tr>
<tr>
<td>316</td>
<td>1399</td>
<td>927</td>
</tr>
<tr>
<td>446</td>
<td>1510</td>
<td>1093</td>
</tr>
<tr>
<td>Hastelloy C</td>
<td>1304</td>
<td>1093</td>
</tr>
<tr>
<td>Hastelloy X</td>
<td>1354</td>
<td>1204</td>
</tr>
<tr>
<td>Incoloy 800</td>
<td>1385</td>
<td>1038</td>
</tr>
<tr>
<td>Inconel 600</td>
<td>1427</td>
<td>1149</td>
</tr>
<tr>
<td>Inconel 625</td>
<td>1427</td>
<td>1149</td>
</tr>
<tr>
<td>Inconel 702</td>
<td>1427</td>
<td>1316</td>
</tr>
<tr>
<td>Inconel X750</td>
<td>1427</td>
<td>1093</td>
</tr>
<tr>
<td>Monel Alloy 400</td>
<td>1349</td>
<td>538</td>
</tr>
<tr>
<td>Nickel</td>
<td>1441</td>
<td>899</td>
</tr>
<tr>
<td>Niobium (Columbium)</td>
<td>2466</td>
<td>538</td>
</tr>
<tr>
<td>Platinum</td>
<td>1771</td>
<td>1760</td>
</tr>
<tr>
<td>Tantalum</td>
<td>3010</td>
<td>399</td>
</tr>
<tr>
<td>Copper</td>
<td>1082</td>
<td>371</td>
</tr>
<tr>
<td>Pt-10Rh</td>
<td>1843</td>
<td>1760</td>
</tr>
<tr>
<td>Pt-13Rh</td>
<td>1843</td>
<td>1760</td>
</tr>
</tbody>
</table>

** Approximate maximum temperature for continuous operation with good resistance to scaling and oxidation.

7.2.2. **Glovebox/Hot Cell Application**

All of the associated difficulties with installation have not been investigated to a great extent. Some of the greatest expected difficulties associated with remote operation are believed to be mounting and measuring thermocouples on a sample. With the current setup, alignment and assembly of the sample column will also be an issue with remote or limited control. To overcome these difficulties, a rigid insulation could potentially be used to hold temperature sensors and the samples in place.

High-temperature, rigid insulations are not common and while having excellent thermal properties, usually do not have great mechanical properties. Various insulations have been
acquired from Zircar Ceramics, Inc., are being experimented with to find out their limitations and potential for use. One of the insulation types holds its dimensions fairly well, while shaping it to functional dimensions is very difficult. Another type that has been experimented with has good characteristics for machining but has an almost chalky makeup. The dimensions don’t hold well with handling and the surface “rubs off.” Work will continue to find a combination of materials that will work as needed.

7.2.3. Thermal Conductivity Modeling

Future work for the project also includes creation and verification of a predictive model for calculating the thermal conductivity of TRISO fuel based on the thermal conductivity values of the constituent materials and the particle volume loading fraction. This work will be a 3 part effort involving physical measurement, theoretical derivation, and computational modeling.

Several models are available in literature for predicting thermal conductivity of different types of heterogeneous materials. Due to variations between the models, model verification samples are needed for measuring the matrix graphite only and the same graphite matrix with a known particle volume fraction. Effort will also be made to effectively model the fuel using finite element methods (FEA) where one challenge needing to be overcome is to accurately model randomly positioned particles at particle volume fractions as high as ~40%.

In addition, considerable effort will be focused on performing microscale measurements of the components of the particles and the interface resistances between the various layers in the particles using laser-based and thermal AFM techniques.
CHAPTER 8
SUMMARY AND CONCLUSIONS

A significant literature review was performed to find the best manner in which to measure the thermal conductivity of a TRISO fuel compact. The guarded-comparative-longitudinal heat flow technique was selected as the ideal method for the compact based on its geometry, size, composition, and thermal conductivity range. Based on the literature reviewed and basic heat transfer principles, a measurement system has been designed. All components of the design system were purchased and/or fabricated. The system has been assembled and is capable of meeting the requirements of the project as measurement results show. Promising results along with analysis of uncertainty shows overall uncertainty to be within 5–10% over a temperature range of 100 to 800°C. A finite element analysis of the system design parameters and operating conditions was performed revealing practical design and operation conclusions.

Some conclusions reached by the initial phase of this project are as follows:

• The most appropriate method to measure thermal conductivity of TRISO fuel compacts is a comparative-axial heat flow method customized to meet the requirements’ specific objectives of this project:

• The TRISO compact thermal conductivity measurement system is:
  - Currently operable over a temperature range of 100 to 900°C

• Capable of measuring samples with thermal conductivities between 10–70 W/m/K. A summary of measured deviations from reference data is shown in Table 8-1.

• The system is capable of measuring samples of composite composition (specifically that of TRISO fuel) as demonstrated by the measured surrogate NGNP graphite/ZrO₂ compact sample. Thermal conductivity values were fairly constant around ~14 W/m/K up to 600°C.
Table 8-1. Resulting deviation from reference values of thermal conductivity of validation samples for given temperature ranges.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$k_s$, W/m/K (100°C-900°C)</th>
<th>$100°C &lt; T_s &lt; 600°C$</th>
<th>$600°C &lt; T_s &lt; 800°C$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inconel 625</td>
<td>10–20 [61-62]</td>
<td>&lt; 6%</td>
<td>N/A</td>
</tr>
<tr>
<td>SS 304</td>
<td>14–30 [57-59]</td>
<td>&lt; 6%</td>
<td>N/A</td>
</tr>
<tr>
<td>Pure Iron</td>
<td>70-30 [59]</td>
<td>&lt; 8%</td>
<td>&lt; 14%</td>
</tr>
</tbody>
</table>

Results measured above 600°C for the graphite/ZrO$_2$ compact are questionable due to thermocouple decalibration (due to very small thermocouple size used).

- Measurement of the NGNP AGR-2 graphite matrix material revealed an interesting phenomenon related to a fundamental change in the material makeup with elevated temperature evidenced by a volumetric change of the sample. The thermal conductivity value permanently increased with temperature above ~450°C. Additional investigation will continue to understand the cause although it seems to be associated with the volume shrinkage experienced by the sample.

- An uncertainty analysis was performed on determinate error in the experimental setup and was estimated to be about ~5.5%. Therefore, based on the results of the validation samples and this analysis, the uncertainty of the measurement system is estimated to be 5-10% for 10 W/m/K < $k_s$ < 70 W/m/K over a temperature range of 100 to 800°C.

- Finite element modeling of the axial-guarded-comparative heat flow method was performed and conclusions reached that are beneficial for this specific measurement system as well as other systems designed using the same method.
  - Low meter bar to specimen length ratio and low specimen aspect ratio are better for reducing systematic error. However, a large distance between temperature monitor positions is helpful in reducing precision error. Thus for a given specimen length, its diameter should be increased.
- Large interfacial thermal resistance increases the system error by blocking axial heat flow; thus the effect of axial heat flow shunting is more significant.

- Matching the guard temperature gradient to the sample temperature gradient provides optimal guard operating conditions while the average guard temperature is much less significant.

- With the success of the first system, the project will continue for the foreseeable future.

  - An entire second setup has been built and is ready to begin testing in order to speed up the data collection process.
  
  - A high temperature system is currently being acquired to extend the temperature range of the measurement and will begin an entire new phase of the project.

- Ultimately a predictive model will be correlated to measured and finite element data based on fuel constituent properties and particle volume fractions.
REFERENCES


