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System to Measure Thermal Conductivity and Seebeck Coefficient for Thermoelectrics

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Abstract

The Seebeck coefficient, when combined with thermal and electrical conductivity, is an essential property measurement for evaluating the potential performance of novel thermoelectric materials. However, there is some question as to which measurement technique(s) provides the most accurate determination of the Seebeck coefficient at elevated temperatures. This has led to the implementation of nonstandardized practices that have further complicated the confirmation of reported high ZT materials. The major objective of the procedure described is for the simultaneous measurement of the Seebeck coefficient and thermal diffusivity within a given temperature range. These thermoelectric measurements must be precise, accurate, and reproducible to ensure meaningful interlaboratory comparison of data. The custom-built thermal characterization system described in this NASA-TM is specifically designed to measure the in-plane thermal diffusivity, and the Seebeck coefficient for materials in the ranging from 73 K through 373 K.

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Nomenclature

TM	technical memorandum
TE	thermoelectric
LLNL	Lawrence Livermore National Laboratory
FoM	figure of merit
σ	electrical conductivity
S	Seebeck coefficient
κ	thermal conductivity
ΔV	voltage difference between the hot and cold sides
ΔT	change in temperature between the hot and cold sides
J	current density
E	intensity of the electric field
R_e	electrical resistance
R_s	sheet resistance
V	voltage
I	current
ρ	resistivity or density
l	length
A	cross-sectional area
W	thermal power
L	length from one end of the sample to the other end
α	thermal diffusivity
c	specific heat
DLC	diamond-like carbon
a	specimen dimension coefficient
Δt	time difference between the maximum hot side and cold side temperature
M	amplitude of the temperature wave on the hot side
N	amplitude of the temperature wave on the cold side
DC	direct current
AC	alternating current
V_H	hot side voltage
V_C	cold side voltage
T_H	hot side temperature

T_C	cold side temperature
TC	thermocouple
GPIB	general purpose interface bus
PID	proportional integral derivative
ZT	dimensionless figure of merit

Overview

A thermoelectric (TE) material possesses the ability to convert thermal energy into electrical energy through a process known as the Seebeck effect, which is based on electron kinetics within differing metallic semiconductors. A thermoelectric module [Figure 1] is a device that is specifically used for the conversion of thermal energy to electrical energy. Within a thermoelectric module, two semiconductors are present, a p-type and an n-type, which are connected thermally in parallel and electrically in series.[1] The p-type and n-type semiconductors are doped to have an excess of positive holes or negative electrons, respectively. The electrical connection between these semiconductors allows for the transfer of electrons from one semiconductor to the other, which enables the flow of current.
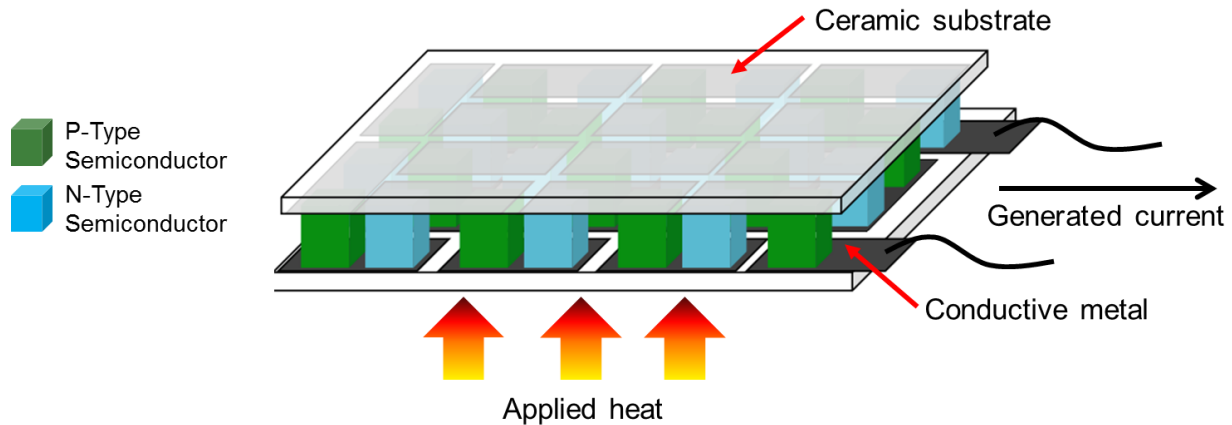
Figure 1 Common thermoelectric module consisting of different semiconductors that are connected thermally in parallel and electrically in series.
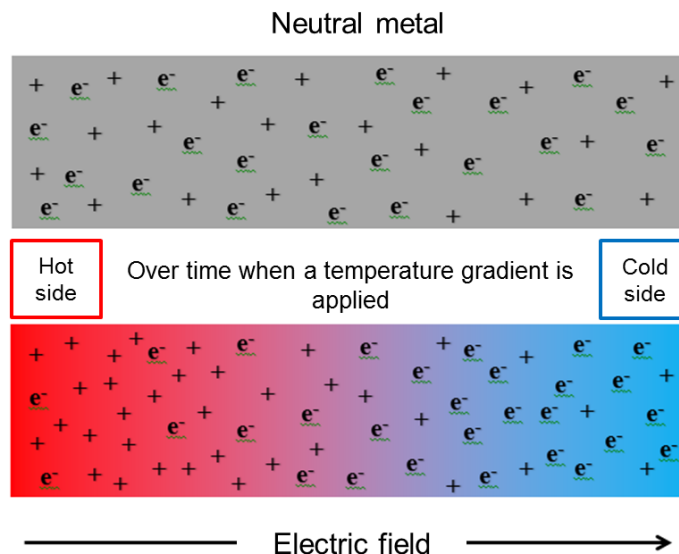
Figure 2 Electron kinetics within a metal before and after a temperature gradient is applied.

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When a temperature gradient is applied to a thermoelectric module (the module is hot on one side and cold on the other) the mobility of electrons is affected. The electrons in the semiconductors on the hot side of the module have more kinetic energy than the electrons on the cold side, due to the presence of thermal energy. Therefore, the cold side of the module obtains excess electrons as they diffuse from an area of higher energy (hot side) to one of lower energy (cold side), leaving an excess of holes at the hot side [Figure 2]. Therefore, when a temperature gradient is applied to a thermoelectric module, a charge gradient is also created, resulting in the formation of an electric field.[2] This process is reversible, converting electrical energy to thermal energy, and is known as the Peltier effect.[3]

Currently, thermoelectric devices still have low conversion efficiencies with ZT = 1.0 – 1.25,[4,5] thereby offering abundant research opportunities that can have a profound impact where thermal energy is abundant. Being conscientious about total energy availability requires full utilization of resources that are present without damaging and wasting them, as is commonly the case. The wasted thermal energy from reactions and processes is no exception. With thermal energy being among the highest sources of wasted energy, waste heat can be extremely beneficial for the utilization of thermoelectric technology. In a 2008 study conducted by the Lawrence Livermore National Laboratory (LLNL), it was found that more energy is wasted than is used [Figure 3].[6] This lack of "energy capture" provides ample evidence as to why thermoelectric technology must continue to be researched and developed.

The National Aeronautics and Space Administration (NASA) has acknowledged the need for thermoelectrics and currently utilizes them as spacecraft and satellite power sources.[7] These spacecraft and satellites rely on or leverage available sources of energy for power such as solar and thermoelectric. The temperature gradient created when a spacecraft or satellite has one side to the sun and one side to open space is significant. Employing thermoelectric technology in space will allow spacecraft and satellites to have an energy source as long as there is a temperature gradient present. However, these technologies are not only useful in space. The push for more sustainable and renewable energy sources has placed thermoelectrics as one of the most promising energy sources for terrestrial applications.[1]
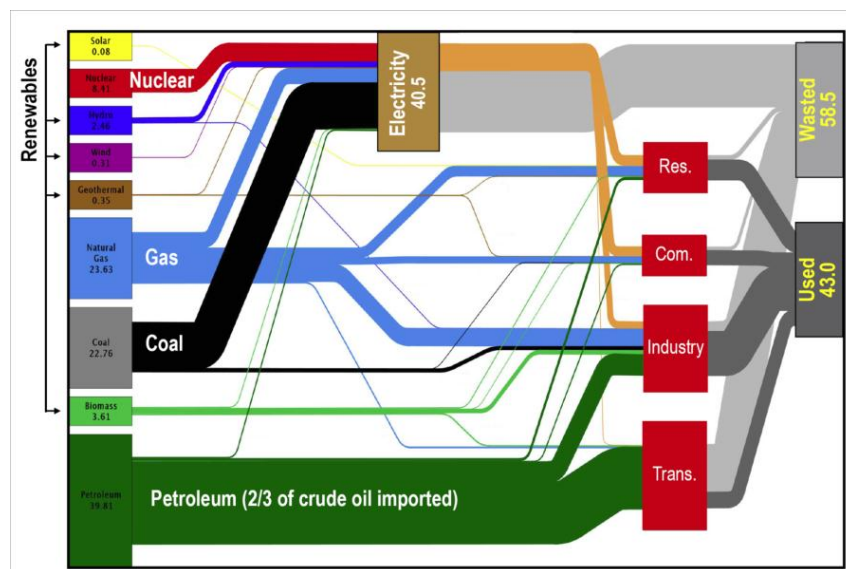
Figure 3 The Lawrence Livermore National Laboratory's finding during their 2008 study of energy usage. [6]

A major objective with thermoelectric technology is to maximize efficiency in order to take advantage of the abundance of waste heat. With this in mind, the effectiveness and performance of a thermoelectric is defined by Equation 1:

$$Z = \frac{\sigma S^2}{\kappa} \tag{1}$$

where Z is the Figure of Merit (FoM), σ is the electrical conductivity, S is the Seebeck coefficient, and κ is the thermal conductivity.

The Figure of Merit (FoM) is a measure of the effectiveness of a thermoelectric, where a higher FoM equates to a higher performance of the thermoelectric (see Table 2 for FoM's of select materials). However, the individual variables that are used in evaluating the FoM must be assessed before the FoM can be calculated and analyzed. These variables are: the Seebeck coefficient, electrical conductivity, and thermal conductivity. Optimization of these variables will lead to a thermoelectric device with a high FoM that efficiently converts thermal to electrical energy (or vice versa).

Principle of determining the Seebeck coefficient

Heat moves through a solid material by means of conduction, the transfer of heat energy by microscopic diffusion and collisions of particles or quasi-particles within a body due to a temperature gradient. The rate of heat transfer per unit area through a material is the heat flux. Heat flux is directly proportional to the temperature gradient, and the constant of proportionality is the thermal conductivity of the material. There are different mechanisms which can transport heat in a material. The first is through vibrations of the internal structure of the material, such as vibrations of the crystal lattices or individual atoms. The other mechanism is through electron transport. In a material that is electrically conductive, electrons can carry heat from one end to another giving rise to the Seebeck effect. A force is needed to stop the drift and maintain equilibrium. This force takes the form of a voltage potential that develops to stop the drift of electrons, and the magnitude of this effect is quantified by the Seebeck coefficient. Seebeck coefficients have units of voltage per unit temperature, usually expressed in microvolts per Kelvin, and can be calculated using Equation 2:

$$S = -\frac{\Delta V}{\Delta T} \tag{2}$$

where ΔV is the voltage difference between the hot and cold sides of a thermoelectric material and ΔT is the change in temperature between the sides. Thermocouples are often used to measure the temperatures ΔT and a voltmeter can be used to measure the voltages ΔV. The ΔV can have a positive or negative value, depending on whether the mobile charges are holes or electrons, thus giving the Seebeck coefficient a positive or negative value.[2]

Principle of electrical conductivity

Electrical conductivity of a thermoelectric can be measured using several methods, the most common being the Hall effect and the four-point probe method. The Hall effect is measured by applying magnetic fields perpendicular to the surface of the sample and electrical currents along the edges of the sample while measuring the voltage across the sample [Figure 4].[8] The electrical conductivity is calculated from Maxwell's Equations for electrical conductivity, as shown with Equation 3:

$$J = \sigma E \tag{3}$$

where J is current density, E is the intensity of the electric field, and σ is electrical conductivity.

The applied magnetic field and current create a force that pushes mobile charges to one side of the material, creating an electric field. This electric field gets stronger until it is equal and opposite of the applied magnetic field.

The Hall effect can be used for other measurements as well, such as bulk concentration [/cm^3], sheet concentration [/cm^2], conductivity [/$\Omega \cdot$cm], and mobility [cm^2/V$\cdot$s] [Figure 4].

Figure 4 A Hall effect experiment (a) experimental set-up of the Hall effect measurement and (b) main test page of the system.

Figure 4(a) shows the experimental set-up of the Hall effect measurement using a compact desktop model with permanent magnets and small circuit system. The Hall effect measurement system is a complete system for measuring the resistivity, carrier concentration and the electron mobility of semiconductors. The system includes software with *I-V* curve capability for checking the ohmic integrity of the user made sample contacts [Figure 4(b)]. The system can be used to characterize various materials including semiconductors and compound semiconductors (n-type and p-type), metals, etc., at both 300 K and 77 K (room temperature and liquid nitrogen temperature) with a funnel for liquid nitrogen.

Electrical conductivity can also be determined using the four-point probe method [9]. This method is conducted by placing four probes (leads) at equidistant intervals in a linear

fashion. Current is supplied through the two outmost probes causing a measurable voltage drop between the two inner probes [Figure 5 (a)]. Using Ohm's law (Equation 4) the resistance R can be calculated.

$$R_e = \frac{V}{I} \tag{4}$$

where R_e is electrical resistance, V is voltage and I is current.

The four-point probe method [Figure 5 (a)] is much more accurate than the similar two-point probe method because separate current and voltage probes are used.[10] In the two-point probe method, the interpretation of the data is more difficult due to the current being passed through the same voltage probes making the voltage drop significant. Whereas in the four-point probe method, the voltage probes have an extremely low (negligible) voltage drop due to the input impedance of the voltmeter, thus giving a more accurate reading.[11]

The van der Pauw technique, due to its convenience, is widely used in the semiconductor industry to determine the resistivity of uniform samples. As originally devised by van der Pauw,[12] one uses an arbitrarily shaped, thin-plate sample containing four very small ohmic contacts placed on the periphery, preferably in the corners, of the plate. This is why the van der Pauw method is usually used, as it is in our case, for Hall effect measurements. A schematic of a rectangular van der Pauw configuration is shown in Figure 5 (b). The objective of the resistivity measurement is to determine the sheet resistance. Van der Pauw demonstrated that there are actually two characteristic resistances R_A and R_B, associated with the corresponding terminals shown in Figure 5 (b). R_A and R_B are related to the sheet resistance (R_s) through the Van der Pauw equation:

$$\exp(-\pi \frac{R_A}{R_S}) + \exp(-\pi \frac{R_B}{R_S}) = 1 \tag{5}$$

which can be solved numerically for R_S.
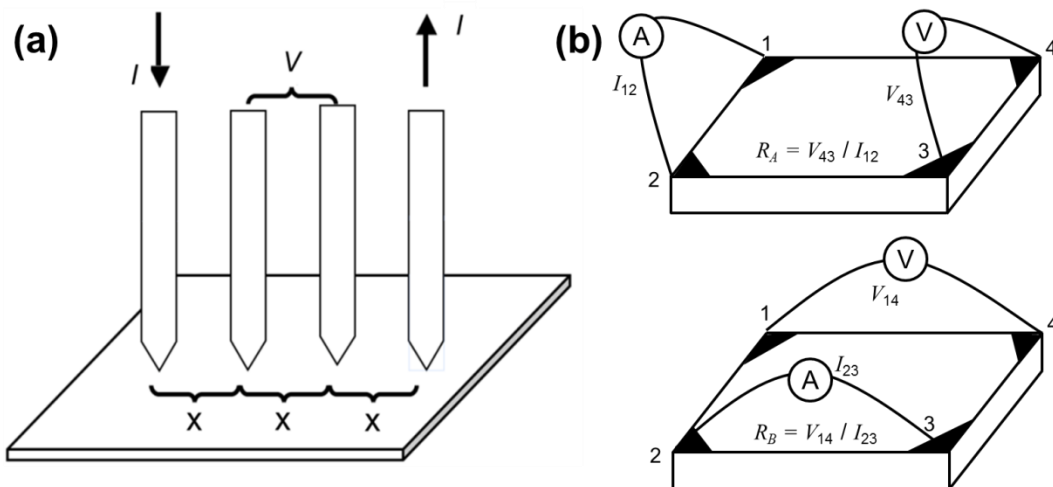
Figure 5 (a) Set-up of the four-point probe method. This method has a higher accuracy than the two-point probe method due to the separate voltage and current probes and (b) schematic of a Van der Pauw configuration used in the determination of the two characteristic resistances R_A and R_B.

Principle of thermal conductivity and thermal conductance

Thermal resistance and thermal conductance of the materials are reciprocals of one another and can be derived from thermal conductivity and the thickness of the materials. Thermal conductivity is the time rate of steady state heat flow through a unit area of a homogeneous material induced by a unit temperature gradient in a linear direction perpendicular to that unit area, W/m·K. Thermal conductance is the time rate of steady state heat flow through a unit area of a material or construction induced by a unit temperature difference between the body surfaces, in W/m²·K.

As with the Seebeck coefficient and electrical conductivity, the thermal conductivity of a thermoelectric can also be measured using different methods. The direct method of thermal conductivity follows Equation 6[13]:

$$W = \kappa \frac{A}{L} \Delta T \tag{6}$$

where W is thermal power, A is the cross-sectional area of the thermoelectric sample, L is the length from one end of the sample to the other end, ΔT is the temperature difference from one end of the sample to the other, and κ is thermal conductivity.

The indirect method of calculating thermal conductivity follows Equation 7:

$$\kappa = \rho \alpha c \tag{7}$$

where ρ is density, α is thermal diffusivity, and c is specific heat at the temperature of interest .[14]

The Figure of Merit for thermoelectrics can be calculated by determining the Seebeck coefficient, electrical conductivity or electrical resistivity, and thermal conductivity. Manipulating the material properties associated with these variables can maximize the FoM to increase the performance level of the material. For instance, by increasing the electrical conductivity while decreasing thermal conductivity would increase the FoM. Typically, thermal conductivity increases as electrical conductivity increases. This poses an obstacle for maximizing the FoM.[15] Finding a way to minimize thermal conductivity while maximizing electrical conductivity, or minimizing electrical resistivity, would produce a thermoelectric of higher performance and greater efficiency.

Reported measurement techniques with chart

Parameter	Publication	Characterization System(s)	Remark(s)
Seebeck coefficient (S)	[16]	The sample is placed between two copper blocks where ΔT and ΔE are measured across the two blocks.	● 10 – 300 K ● Through the thickness ● Polycrystalline pellets
	[17]	The potential difference and change in temperature are measured across a heat source and heat sink with the sample in the middle.	● 150 – 300 K ● Through the thickness
	[18]	Thermocouples are used to measure the temperature and voltage difference.	● 300 – 1300 K ● Bulk ● In-plane
	[19]	The potential difference and change in temperature are measured across a heat source and heat sink with the sample in the middle.	● 296 – 1200 K ● Bulk ● Through the thickness
Electrical Conductivity (σ)	[20]	Small electron or ion beam deposited strips can be connected to larger metallic pads for probe readings if they are too small. Probing a metallic deposition strip on an insulating background gives a direct measurement method.	● Temperature not specified ● In-plane
	[21]	Two-point probe method with an electrometer is used for measurements.	● 300 – 550 K ● Through the thickness
	[22]	Van der Pauw Method is used with a nanovoltmeter to measure conductivity and temperature.	● 10 – 300 K ● Through the thickness ● Polycrystalline pellets
	[23]	Mobility and conductivity are calculated from Hall effect measurements.	● 20 – 500 K ● In-plane
	[24]	A modified Wheatstone bridge and AC current are used.	● 150 – 300 K ● Through the thickness
Thermal Conductivity (κ)	[25]	Thermal conductivity is measured by the transient technique using a line heat source.	● 6 – 20 °C ● Through the thickness
	[26]	Thermal conductivity is calculated using known heat flows, equations, and assumptions about the heat flow.	● 278 – 293 K ● Through the thickness

	[27]	The Laser Flash method involves heating a homogenous material with a laser while measuring the temperature versus time.	● 77 and 300 K ● Through the thickness
	[28]	A current is sent through a metallic strip at a certain frequency to heat the sample and the thermal conductivity is evaluated by measuring the frequency dependence of the temperature oscillations.	● 35 – 500 ^{o}C ● Through the thickness
	[29]	An alternating heat pulse is applied at one end of a metal rod while leaving the other end at room temperature. The thermal conductivity can be measured using the temperature at two points on the metal rod that are measured as a function of time.	● Temperature not specified ● In-plane

The following are the previously reported best methods for measuring the Seebeck coefficient and thermal conductivity, which are measured by our TE characterization system. Electrical conductivity is not discussed from here because our TE system does not measure this parameter. Therefore, in order to calculate the ZT factor, it must be measured using the Hall effect system (see Figure 4).

Reported Seebeck coefficient system

The Seebeck coefficient of a thermoelectric material can be measured by placing the material in between two conducting metal blocks, such as copper. One end of the sample is heated while the other acts as a heat sink, dispersing the heat, thus cooling that side. Thermocouples are then placed on the thermoelectric material, between the metal blocks. This set up for the measurement of the Seebeck coefficient is one of the simplest and most widely used. When one side of the module is heated up the thermocouples register the temperature difference in the material. This change in temperature produces a detectable voltage drop, which is measured with the thermocouples as well. The measurements are made for different ambient temperatures that relate to the operating range of the thermoelectric.

Reported thermal conductivity system

Currently, there are two primary methods for determining the thermal conductivity of a material. One is the Flash method, which uses a high energy pulse to deliver a short burst of energy to the sample while measuring the temperature on the opposite side. The other method is called the Three Omega method and is an adaptation of Angstrom's method. Both the Three Omega and Angstrom's methods use a periodic heat source to generate a heat wave in the sample. The methods then measure the amplitude decay and the phase shift of the heat wave as it travels through the sample. The Flash method is a very fast test that measures the thermal conductivity through the thickness of a sample. This works for a bulk material, but a thin ceramic or metallic film depends strongly on the orientation so it should be tested along its crystal axes. The noticeable disadvantage of the Flash method is that it does not allow for testing of the

Seebeck coefficient because the test occurs so quickly that the temperature differences are too small to gather meaningful data for the Seebeck coefficient. Angstrom's method held some promise, but commercially available systems altered the sample, possibly changing its thermal characteristics.

Motivation of TE characterization system setup

The Seebeck coefficient, when combined with thermal and electrical conductivity, is an essential property measurement for evaluating the potential performance of novel thermoelectric materials. However, there is some question as to which measurement technique(s) provides the most accurate determination of the Seebeck coefficient at elevated temperatures. This has led to the implementation of nonstandardized methods that have further complicated the confirmation of reported high ZT materials. A major consideration for the procedure described is for the simultaneous measurement of the Seebeck coefficient and thermal diffusivity within a temperature range. These thermoelectric measurements must be reliable, accurate, and consistent to ensure meaningful interlaboratory comparison of data.

Practical design criteria and performing calculation

Chamber, temperature control, and sample holder

Thermal conductivity is a function of temperature, so data must be gathered across a range of temperatures. The standard range is from about 73 K (-200 ^{o}C) to 373 K (100 ^{o}C). Two systems are needed to gather data for this entire range: one to go from cryogenic to room temperature and another to go from room temperature up to 373 K. The two systems should be similar in design so that the method is consistent throughout the range. The cryogenic chamber is a highly confined space, so all components of the system are required to be small and perform in a vacuum. The system must operate in a vacuum because at cryogenic temperatures any air or moisture in the chamber will condense on the sample. The contact probes must be made of a material that minimizes heat conduction, i.e. has a low thermal conductivity. Accurate measurements must be taken quickly, and be corrected for any offsets that may occur such as the Seebeck effect of the probes in the voltage measurements. The selected system closely resembles other systems that use Angstrom's method. The sample is mounted vertically with a heater attached to an arm and resting on the cold plate. The probes are mounted in a spring assembly and can be moved in and out to achieve the desired pressure. Temperature is measured using T-type thermocouples and voltage is measured using a lead from each thermocouple. The heater is a three watt 1000 Ω resistor attached to the specimen clamp. Data are gathered using three multi-channel Keithley nanovoltmeters and the raw data is processed via a computer program [see Figure 6].

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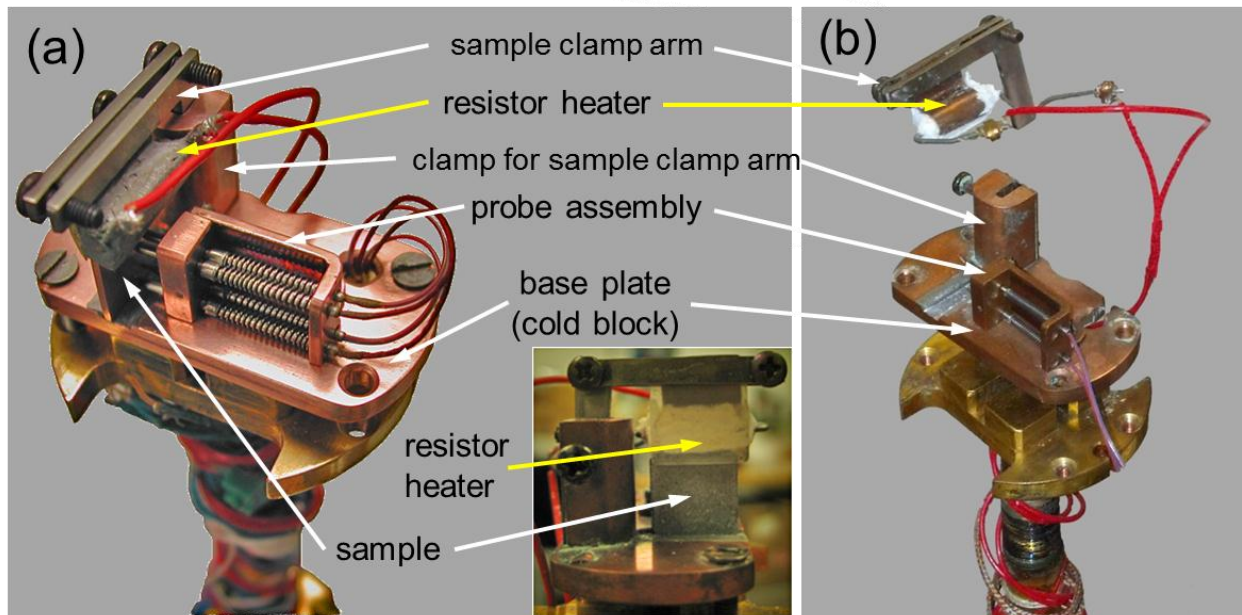
Figure 6 (a) Thermal characterization system and (b) an exploded view of (a).

Seebeck coefficient

The program records the temperature and voltage differences versus time, then uses these values to compute the thermal diffusivity and the Seebeck coefficient. The Seebeck coefficient is calculated in one of two ways depending on the method that the data were gathered. If the entire loop was gathered (both the heating and cooling sides of the curve) then linear fits for the heating and cooling sides of the curve are computed and averaged to find the best possible fit. If only one side of the curve is gathered, then a linear fit for the voltage versus temperature data is calculated where the slope of the fit is the Seebeck coefficient. Theoretically, the voltage difference should vary linearly with the temperature difference on both the heating and cooling phases, but this is not always so because the sample is changing temperature as the test progresses. Hence the Seebeck coefficient changes resulting in a hysteresis loop instead of a linear plot.
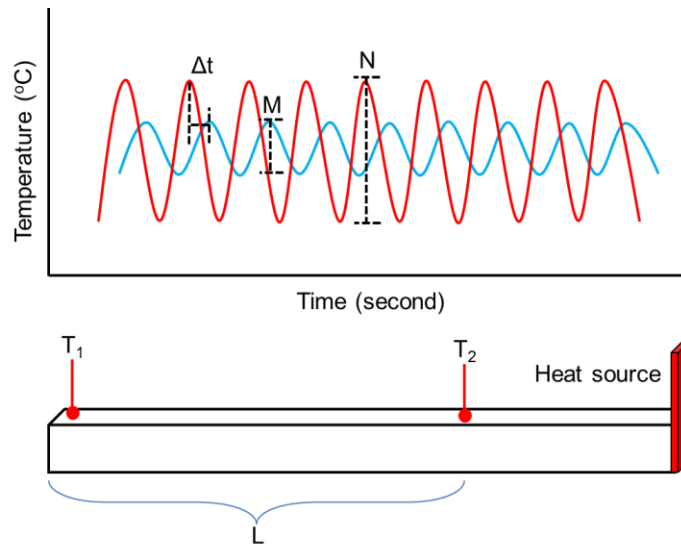
Thermal diffusivity

The thermal diffusivity is calculated using the temperature differences versus time measured by the thermocouples, specifically the maximum temperature reached by both the cold and hot sides and Equations 8 and 9:

$$\alpha = \frac{L^2}{a\Delta t \ln\frac{M}{N}} \tag{8}$$

$$\kappa = \alpha\rho c \tag{9}$$

where α is the thermal diffusivity, L is the length between the thermocouples, a is the specimen geometric dimension coefficient that depends upon the nature of the setup and sample shape, Δt is the time difference between the maximum hot side temperature and the maximum cold side temperature, M is the amplitude of the temperature wave on the hot side and N is the amplitude of the temperature wave on the cold side [Figure 7]. Equation 8 comes from Angstrom's method and requires the user to input both L (by measuring the actual distance between the probes) and a (by testing a known sample). Calibration of the system requires selection of a value for L that is the actual distance between the hot and cold probes, then testing a known sample and adjusting a so that the measurement is correct. The values for L and a should be checked against another known sample with a different thermal conductivity. M is the maximum change in the hot temperature, and N is the maximum change in cold temperature. The time between the two maximums is Δt. The formula for the thermal conductivity works well as long as there is a well-defined peak in the temperature graphs.


Figure 7 Physical definitions of the variables within the equation of thermal diffusivity; (a) typical data plot for thermal conductivity measurement and (b) setup for thermal diffusivity measurement.

The thermoelectric characterization system setup with fixture design

The thermal characterization system in this NASA-TM is designed to measure the in-plane thermal diffusivity and the Seebeck coefficient for materials in the range of temperatures from 73 K through 373 K. As shown, the system consists of a resistance heater mounted on an adjustable sample clamp arm which is attached to the base plate (cold block). This resistance heater gives a thermal pulse to the specimen that exceeds the ambient temperature. The geometry of the sample should be square-shaped, approximately 1 cm × 1 cm, with all opposite faces parallel, to mount vertically with a heater attached to an arm and resting on the cold plate. If the sample is a thin film on a substrate, the substrate should have a thermal conductivity a few orders of magnitude lower than the sample to prevent the substrate effect. A probe assembly, mounted to the base plate, simultaneously measures the hot and cold temperatures and the Seebeck voltage from a heat pulse traveling from the resistance heater to the colder base plate.

The system developed here uses three Keithley 2182A nanovoltmeters for simultaneous measurements of the thermal diffusivity and Seebeck coefficient. A Keithley 6221 DC and AC current source is used to apply power to a high power resistor. The nanovoltmeters and the current source are connected to a computer with GPIB interface. A schematic diagram of the system is shown in Figure 12. When a thermal pulse is created within a sample by a DC/AC current source, the thermal pulse is transported by both energetic electrons and phonons through the sample to the colder base plate. Two thermocouples that are placed on a sample with a certain gap distance measure temperatures instantaneously to identify the thermal pulse that passes through the specimen. The temperature difference between the probes and the time interval on which it occurs will indicate the thermal diffusivity of the sample material. The thermal conductivity can then be calculated from the thermal diffusivity data if the density and specific heat are known. As shown in Figure 8, two voltage probes are almost collocated with two thermocouples in proximity to measure the Seebeck coefficient which is an intrinsic property of a material. The voltage difference between two locations (or two temperatures at two locations) indicates the electrostatic potential associated with temperature and is called the Seebeck coefficient.
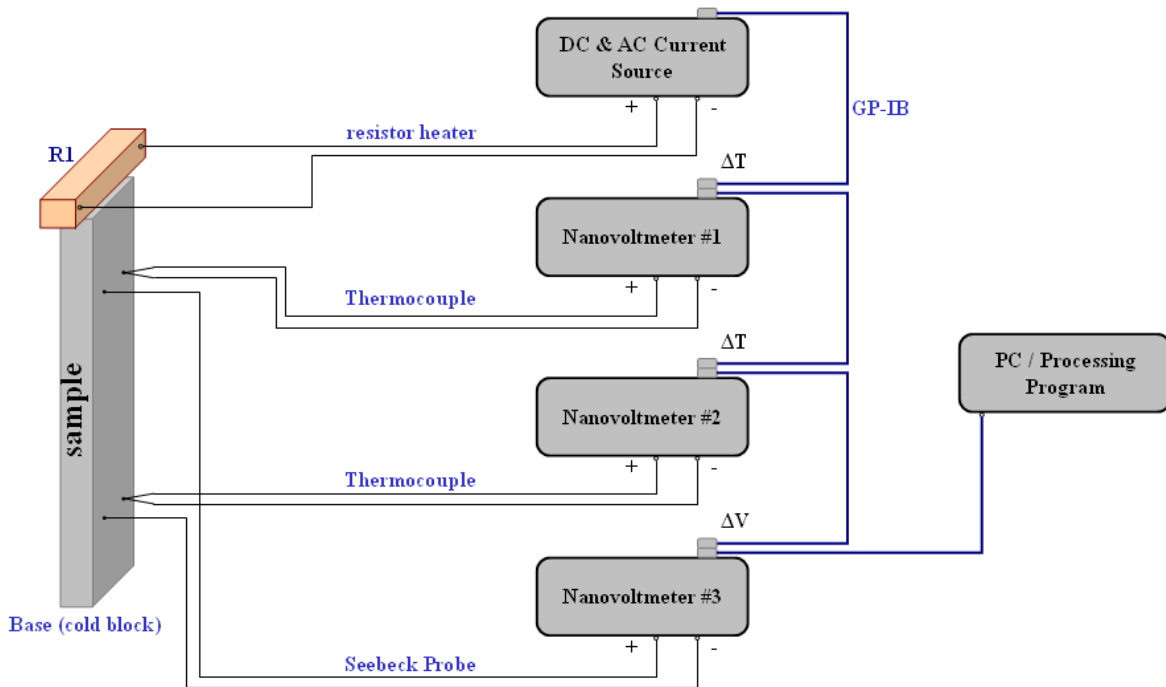

Figure 8 Thermal characterization system schematic diagram.

These two important parameters, the thermal conductivity and Seebeck coefficient, are the key factors to determining the FoM of thermoelectric materials. The system itself is configured for the measurements of these two parameters from cryogenic to high temperatures. There are no such systems available to cover an entire temperature range. The unique nature of this system is to measure the thermal conductivity and Seebeck coefficient simultaneously.

The probe assembly [Figure 9] houses the hot and cold side voltage probes (V_H and V_C) for measuring Seebeck voltages and the thermocouples (T_H and T_C) for measuring temperature difference and thermal diffusivity (see Figure 7). All probes are fabricated with stainless steel

tubing and collars with pressure applied to the sample with tungsten wire springs. The dimensions and material type may be altered without affecting the assembly function. The probe spacing can also be varied in the vertical direction for compatibility with the sample material and heat source, although the horizontal spacing should be as small as possible. The voltage probes are fiberglass-wrapped copper wire bonded in place with a ceramic compound, such as Cotronics Resbond 919. This compound has a low thermal conductivity. The thermocouple probes are similarly made using T-type thermocouples, although other types may be used depending on the temperature range of interest.
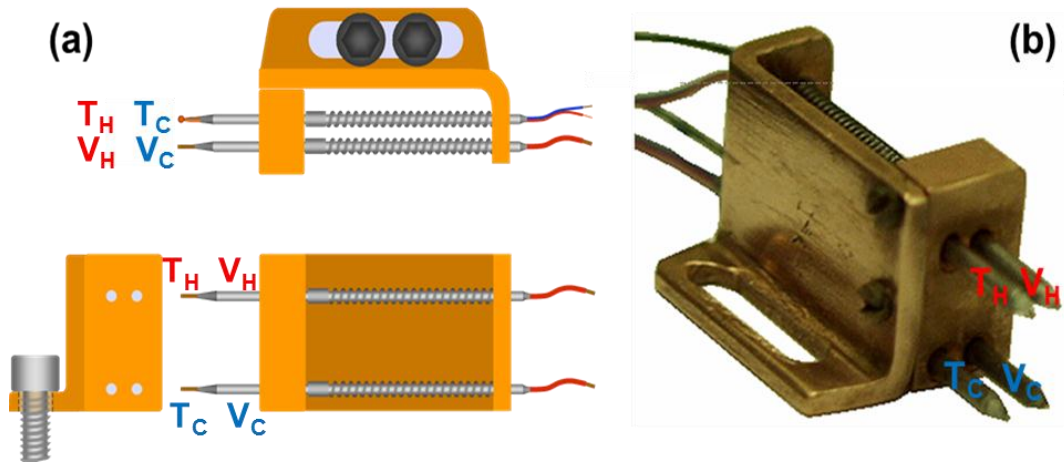
Figure 9 Probe assembly; (a) schematic diagram for probe housing and (b) copper probe housing.

The sample clamp arm assembly consists of a copper support post, two piece stainless steel support arm, and a resistor heater. The support post [Figure 10] is made of copper for dissipating the heat generated from the resistor heater after the heat pulse is applied to the sample, (see Figure 7). A bolt threaded in the side secures the support arm and allows different lengths of samples to be tested. For samples with a greater distance between the hot and cold temperature measurements, it will be necessary to extend the support arm height for the longer sample.
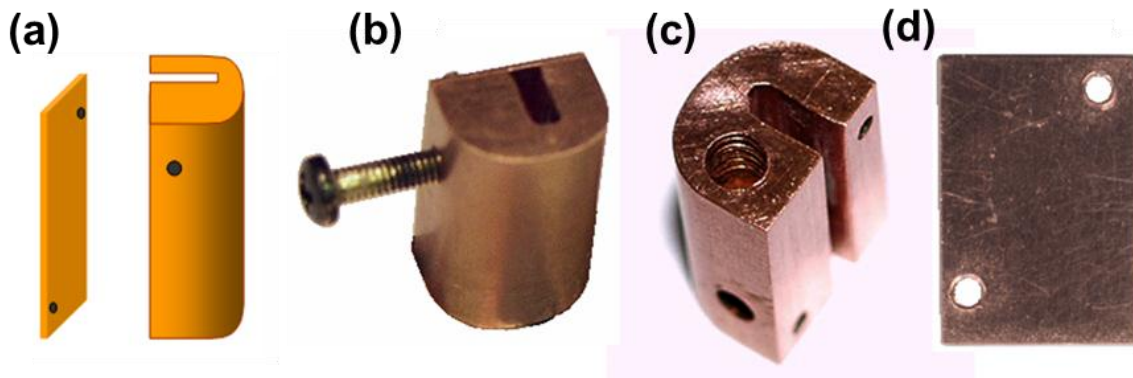
Figure 10 Sample clamp arm; (a) schematic diagram, (b) top view, (c) bottom view, and (d) support post with back plate.

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The support arm [Figure 11] is made of stainless steel for directing the energy of the initial heat pulse through the sample and for dissipating the residual heat through the support post. The two pieces clamp the sample heater assembly (see Figure 6).
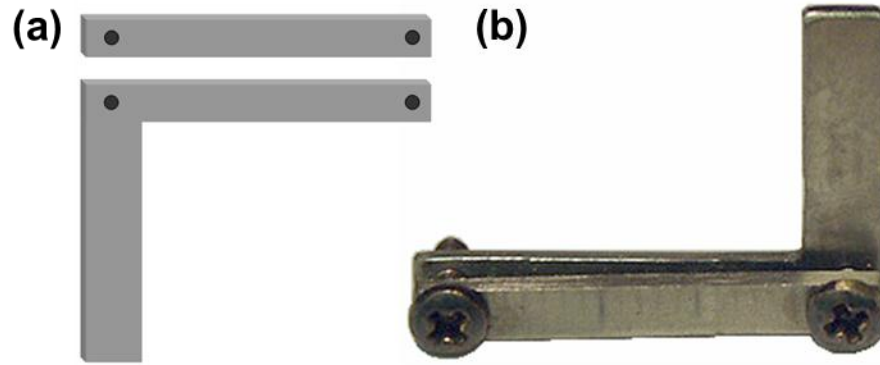

Figure 11 Sample clamp arm with support arm; (a) schematic diagram and (b) assembled, side view.

The heater assembly, shown in Figure 12, consists of a resistor heater attached to a sapphire sheet with thermally conducting, electrically insulating ceramic compound (see Figure 6). The sapphire conducts the residual heat through the support arm after the heat pulse is applied to the test sample. The source of the applied heat pulse can be a resistor, coil heater, laser diode, or similar device. It should apply heat evenly along the entire width of the sample. Lead wires for supplying current should be able to withstand the ambient temperature of the sample during testing in addition to the heat generated from the heater. Sample measurements are taken at several increments within the ambient temperature range of approximately 73 K through 373 K. For slow ambient temperature ramp times, the measurements may be taken in a continuous mode while the ambient temperature is changing as long as the ambient temperature change is accounted for in the calculations. Alternatively, the ambient temperature for each measurement increment can be allowed to stabilize before taking the measurement. The second method will also allow more than one measurement to be taken and averaged at each ambient temperature increment.
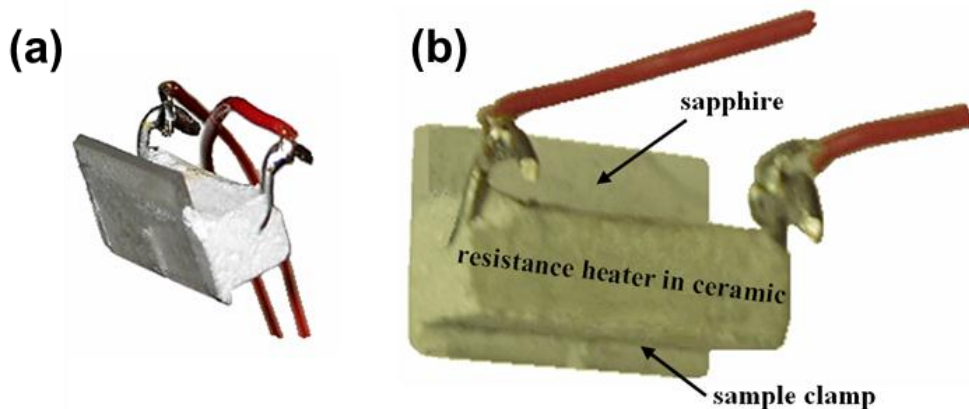

Figure 12 Heater assembly; (a) side view and (b) back view with resistance heater in ceramic.

The entire thermal characterization test fixture can be used in both a cryogenic chamber and a vacuum furnace. The probe assembly is removable to allow a different thermocouple type to be used for the temperature range of interest and to allow a different vertical probe spacing to permit samples in a wide range of thermal diffusivities to be tested.

The base plate, Figure 13, (see also Figure 6) functions as a heat sink (cold block) in the measurement system. A shallow groove in the plate holds the sample against the probes. The groove must be coated with electrical insulating compound. Electrical isolation of the entire sample is required to isolate the probe measurements.
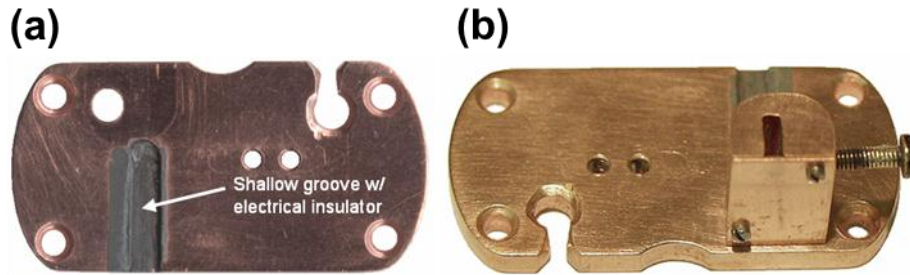
Figure 13 Base plate; (a) top view without support post and (b) back view with support post.

The characterization system was custom-built to accommodate specific design details. A picture of the TE characterization system is shown in Figure 14 that is comprised of a vacuum system, temperature controlled oven, and a semi-automatic data acquisition system. Thermal measurement control and output is accomplished through computer software. A LabVIEW® software program can be used to apply the heater current for a user-designated pulse time and to record the thermal diffusivity and the Seebeck coefficient.
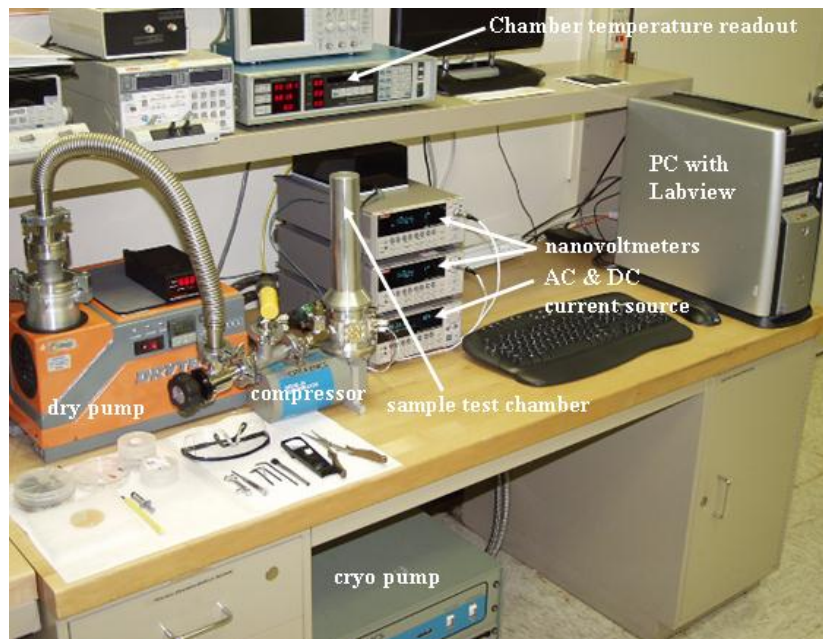
Figure 14 The low temperature TE characterization system.

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Program logic to measure thermal conductivity and Seebeck coefficient

Custom-designed software was written to allow for semi-automatic data acquisition and analysis [Figure 15].
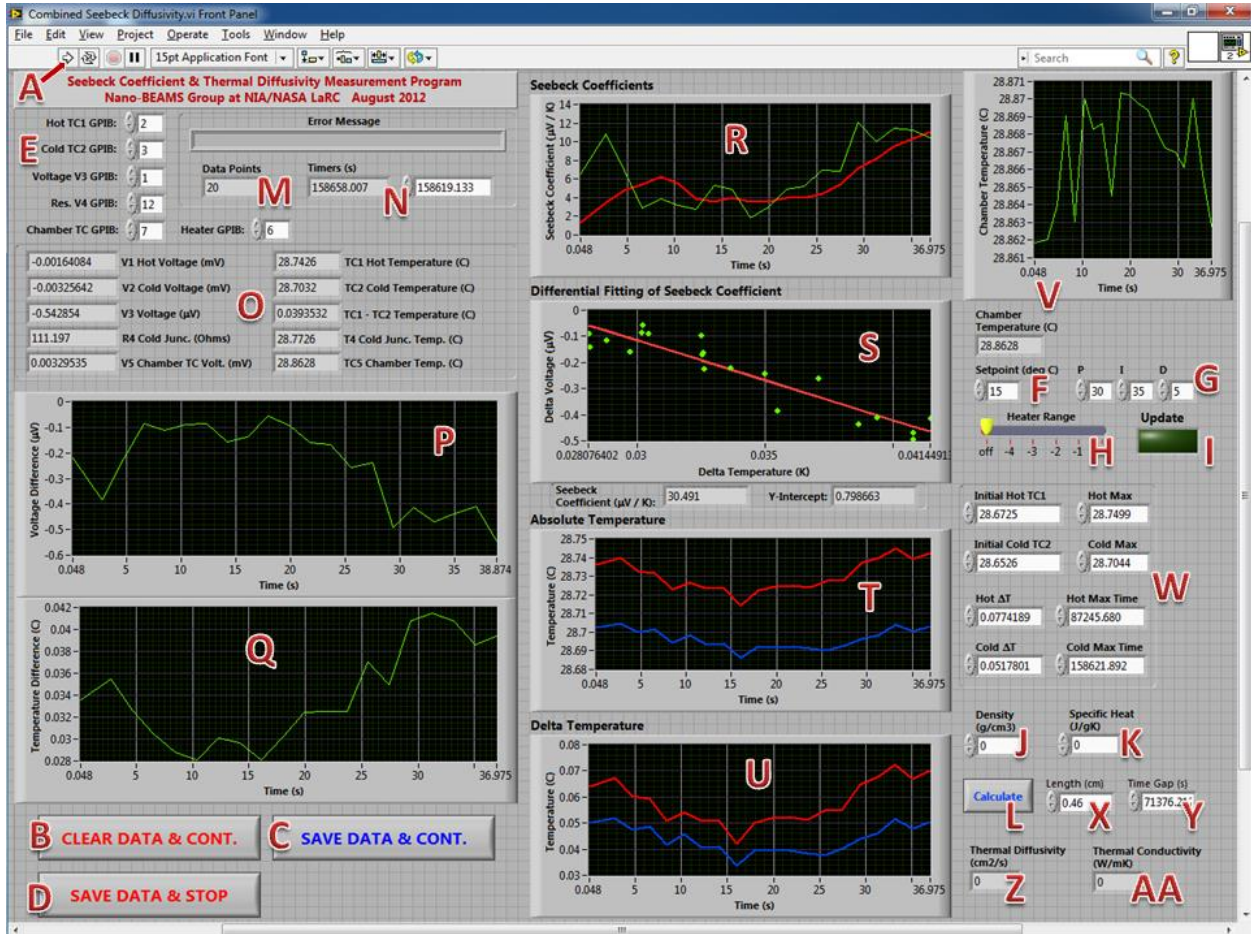
Figure 15 LabVIEW® graphical user interface

Description of Front Panel controls and their use in the program

A) LabVIEW® Run – "Click" to depress this button and begin the LabVIEW® program. A message will be displayed and the program will not run if an error is encountered.

B) Clear Data & Continue – This button clears the data in memory, initializes parameters, and then continues to take measurements.

C) Save Data & Continue – This button allows the data in memory to be saved before clearing it (data are never automatically saved). Measurements continue after the data are saved and cleared.

D) Save Data & Stop – This button allows the data in memory to be saved before stopping the program.

E) GPIB Addresses – Variable inputs for GPIB addresses of the hot side thermocouple (Hot TC1), cold side thermocouple (Cold TC2), voltage difference between the thermocouples (Voltage V3), cold junction resistance temperature detector (Res. V4), chamber thermocouple (Chamber TC), and the heater (Heater).

F) Setpoint – Selection of the temperature setpoint for the chamber.

G) PID Tuning Parameters – Sets the tuning parameters (proportional, integral, derivative) for the heater's temperature controller.

H) Heater Range – This controls the maximum heater output.

I) Update – This button executes the values entered for the Setpoint, PID tuning parameters, and the Heater range.

J) Density – The user may enter the sample density for calculation of the thermal conductivity.

K) Specific Heat – The user may enter the specific heat for calculation of the thermal conductivity.

L) Calculate – This button will calculate the thermal diffusivity (and possibly thermal conductivity) from the acquired data.

Indicators and what user is measuring

M) Data Points – Displays the total number of data points saved in memory.

N) Timers – The absolute computer clock time, which is used to assign the relative time at which data are taken. Measurements begin at time equal to zero.

O) Real-time Measurement Parameters – These display the real-time values of the hot (V1) and cold (V2) thermocouple voltages, the hot (TC1) and cold (TC2) thermocouple temperatures, the hot and cold thermocouple temperature difference (TC1 – TC2), the voltage difference between the hot and cold thermocouples (V3), the cold junction resistance (R4), the cold junction temperature (T4), the chamber thermocouple voltage (V5), and the chamber thermocouple temperature (TC5).

P) Voltage Difference Plot – The measured voltage difference between the hot (TC1) and cold (TC2) thermocouples is plotted versus relative time.

Q) Temperature Difference Plot – The measured temperature difference between the hot (TC1) and cold (TC2) thermocouples is plotted versus relative time.

R) Seebeck Coefficient Plot – The instantaneous (green curve) and average (last five measurements, red curve) Seebeck coefficients are plotted versus relative time.

S) Differential Fitting of Seebeck Coefficient Plot – The difference in voltage versus the difference in temperature of the hot and cold thermocouples is plotted (green data). A linear fit to these data is displayed and the Seebeck coefficient using differential fitting is calculated (red curve) and displayed numerically.

T) Absolute Temperature Plot – The absolute temperature of the hot (TC1) and cold (TC2) thermocouples versus relative time is plotted.

U) Delta Temperature Plot – The change in temperature from the initial temperature of both the hot (red curve) and cold (blue curve) thermocouples versus relative time is plotted.

V) Chamber Temperature Plot – The temperature of the chamber thermocouple versus relative time is plotted and displayed numerically.

W) Thermal Diffusivity Parameters – Numerical displays for the initial temperatures of the hot (TC1) and cold (TC2) thermocouples, their maximum temperatures and the time at which they occurred, and the temperature difference between the maximum and initial values.

X) Length – The distance between the hot (TC1) and cold (TC2) thermocouples.

Y) Time Gap – The time elapsed from the maximum temperature of the hot (TC1) thermocouple to the maximum temperature of the cold (TC2) thermocouple.

Z) Thermal Diffusivity – This value is the calculated thermal diffusivity.

AA) Thermal Conductivity – The thermal conductivity is calculated if the sample density and specific heat are known.

Calibrating the system

In our design, the distance between the probes is always the same, so the length can be measured to within a few tenths of a millimeter with a micrometer and then any small adjustments that need to be made can be accomplished by adjusting the constant "a" to fit the known samples (see Equation 8). The known samples should be from the expected range of use. Since the primary use of the system is testing thermoelectric materials, and these materials tend to have low thermal diffusivities, an insulator like glass makes a good calibration sample. At the other end of the spectrum, graphite is a good choice because it is available as a calibration sample, and it has high thermal conductivity so it is at the very edge of the system's range.

DATA comparison (with our system and NETZSCH LFA 457 MicroFlash[®]

Table 1 Calculated and tested thermal diffusivity with graphite, quartz glass, silicon wafer, and 2948 glass slide with NASA Langley Method at room temperature.

Material	Published data (a)			Calculated thermal diffusivity (cm²/s) (b)	Tested thermal diffusivity with the system (cm²/s) (c)
	Specific heat (J/kg)	Density (kg/m³)	Thermal conductivity (W/m·K)		
Graphite	714.34	1730	1730	0.7388	0.8
Quartz glass	745	2200	1.381	0.0084	0.0083
Silicon wafer	703	2330	125.52	0.7663	0.756
2948 glass slide	753	2300	1.399	0.0077	0.007

(a) Reference [30]

(b) $\kappa = \alpha \rho c$ (equation 9), where κ is the thermal conductivity, α is the thermal diffusivity, ρ is the density, and c is the specific heat of the sample.

(c) Data from NASA Langley's Instrumentation

Table 2 Tested data comparison of quartz and micro-scale Bi$_2$Te$_3$ grains covered with 0.5 wt% and 0.05 wt% Au samples with ASTM E1461 Flash Method [31] and NASA Langley's Instrumentation

Netzsch Data
ASTM E1461 Flash Method Thermal Conductivity Results

Sample	Thickness @25°C [mm]	Bulk Density ρ@25°C [kg/m³]	Temperature [°C]	Specific Heat C_p [J/(kg·K)]	Diffusivity α [m²/s]	Thermal Conductivity λ [W/(m·K)]	Seebeck Coefficient S [μV/K]	Electrical Conductivity σ [1/(Ω·cm)]	Z [/K]	Figure of Merit ZT [300K]
Quartz Glass	0.965	2193	25	733.4	8.20E-07	1.32	---	---	---	---
			100	849.7	7.79E-07	1.45	---	---	---	---
			200	959.7	7.40E-07	1.56	---	---	---	---
			300	1044.1	7.20E-07	1.65	---	---	---	---
Bi$_2$Te$_3$ 10-1 (0.5 wt% Au)	0.503	7209	25	208.6	7.94E-07	1.19	181.00	1355.70	0.0037	1.116
			100	225.1	7.42E-07	1.20	181.00	1355.70	0.0037	1.106
			200	232.9	7.84E-07	1.32	181.00	1355.70	0.0034	1.012
			300	230.4	7.41E-07	1.23	181.00	1355.70	0.0036	1.083
Bi2Te3 10-3 (0.05 wt% Au)	0.581	7655	25	182.8	9.96E-07	1.39	181.00	1355.70	0.0032	0.956
			100	187.9	9.14E-07	1.31	181.00	1355.70	0.0034	1.014
			200	193.5	9.50E-07	1.41	181.00	1355.70	0.0032	0.947
			300	194.7	1.09E-06	1.62	181.00	1355.70	0.0027	0.821

LaRC Data

Sample	Thickness @25°C [mm]	Bulk Density ρ@25°C [kg/m³]	Temperature [°C]	Specific Heat C_p [J/(kg·K)]	Diffusivity α [m²/s]	Thermal Conductivity λ [W/(m·K)]	Seebeck Coefficient S [μV/K]	Electrical Conductivity σ [1/(Ω·cm)]	Z [/K]	Figure of Merit ZT [300K]
Quartz Glass	N/A	2187	25	744.8	8.68E-07	1.41	---	---	---	---
Bi$_2$Te$_3$ 9-2		5780		163	1.13E-06	1.06	163.00	1205.10	0.0030	0.902
Bi$_2$Te$_3$ 9-3		5780		133.25	1.33E-06	1.02	181.00	1205.10	0.0039	1.156
Bi$_2$Te$_3$ 9-4		5780		133.25	1.33E-06	1.02	181.00	1400.00	0.0045	1.343
Bi$_2$Te$_3$ 10-1 (0.5 wt% Au)	N/A	7052	25	133.25	1.31E-06	1.23	181.00	1355.70	0.0036	1.086
Bi$_2$Te$_3$ 10-3 (0.05 wt% Au)	N/A	7511	25	133.25	1.37E-06	1.37	181.00	1355.70	0.0032	0.973
		7511		82.8	1.37E-06	0.85	181.00	1355.70	0.0052	1.564

NOTES:

1. C_p for quartz glass is a database reference (from Thermtest Corp).

2. C_p for both LaRC Bi$_2$Te$_3$ samples are measured in powder form from other 0.05 wt% sample.

3. Netzsch measures the C_p using a solid sample during the Flash Method.

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Appendix (Manual): Work instructions for Thermal Characterization

[1] Preparing the sample

- The geometry of both bulk and thin film samples should be square-shaped, approximately 10 mm × 10 mm. Although, samples between 5 mm and 20 mm and up to 3 mm in thickness are acceptable.

[2] Mounting the Sample

- The bottom of the sample rests in a groove in the cold plate.

- The top of the sample is held in place by the heater arm.

- Secure the heater arm using the screw in the upright section of the arm assembly. Pliers should be used as the head of the screw cannot be reached.

- After securing the sample bring the probe assembly into contact with the sample and apply pressure so that all the probes contact the surface. The probes are on springs so make sure that each probe is in contact with the sample.

- Secure the probe assembly using the two hex screws at its base.

[3] Evacuating the chamber

- Place the chamber cover on the chamber and secure it with four hex screws with washers.

- Ensure that the vent valve is closed.

- Open the valve at the end to connect the chamber to the vacuum pump.

- Turn on the vacuum pump.

- Let the chamber reach a vacuum of at least 5 mTorr.

[4] Running the Test

- Open the program "Combined Seebeck Diffusivity" in the Shared Documents folder.

- At the opening screen, set the following values:

  o GPIB Addresses (default values should be correct)
  o Setpoint Temperature of Chamber (room temperature)
  o PID Tuning Parameters
  o Heater Range (off).

- Press Run – Starts the program.

- Once measurements are stabilized, press Clear & Continue to dump the previous data and begin measurements.

- To end a test, press Calculate, and then press Save & Continue to save the data in an array.

- To exit the program, make sure that the heater is off, and press the Save & Stop or the Stop button.

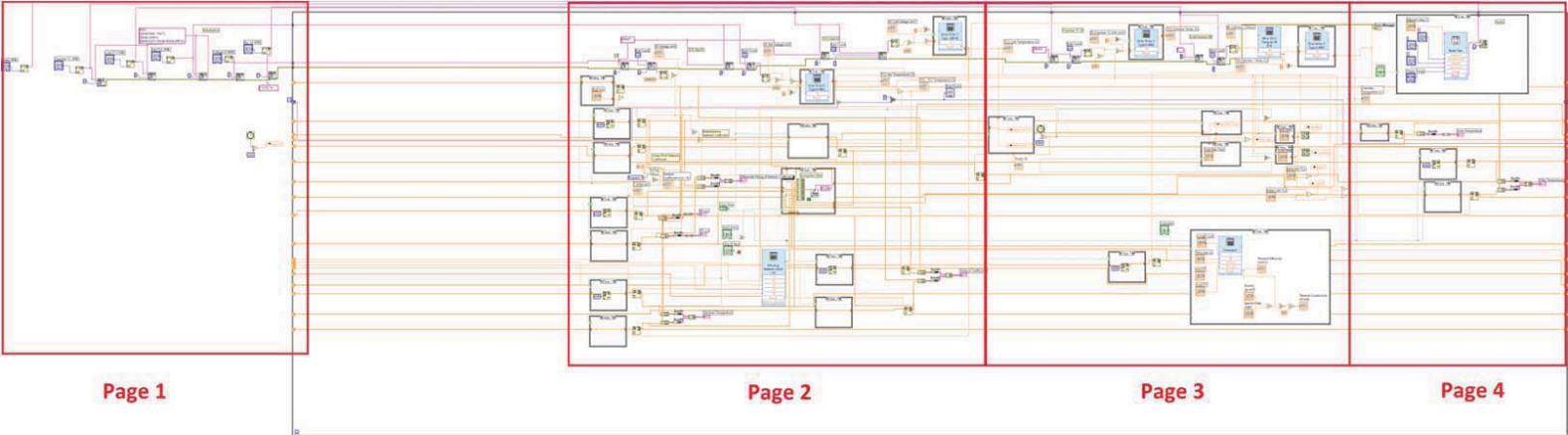**[5] Internal Program Operation**

- Program initiation:

Various instruments are initialized when the program first starts. These include the nanovoltmeters for thermocouples (chamber, hot, cold) and the potential difference between the hot and cold thermocouples, the cold reference junction resistance temperature detector, and the heater. The beginning time is also initialized.
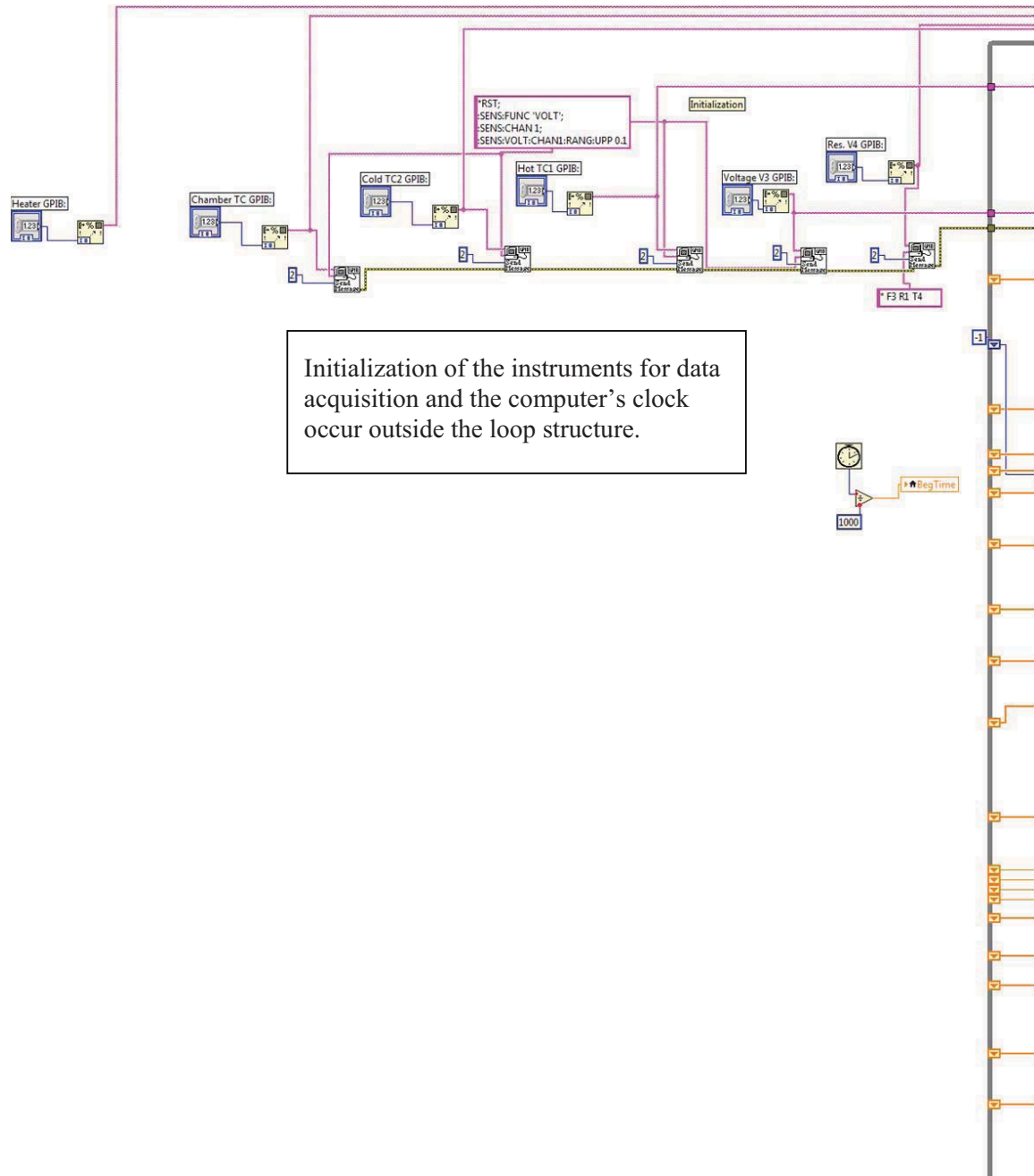
- Loop:

After initiation, the program enters a while loop, which is the main loop for the program. It will repeat until the stop button is clicked. Inside the loop, data are acquired from the various instruments, displayed numerically, plotted in graphs, and used to calculate certain parameters. These data, graphs, and numerical displays are described under the section on front panel controls. User supplied inputs while the loop is running can be altered; these include clearing or saving data, changing the chamber temperature, changing the heating parameters, and calculating thermal parameters. These raw and computed data are saved in a two-dimensional array that grows as the loop iterates and can be saved by the user to a specified filename.

The block diagram of the LabVIEW® program is shown below in full, along with magnified sections of the code.

The LabVIEW® block diagram below is enlarged and sectioned on the next four pages.

Page 1   Page 2   Page 3   Page 4

Heater GPIB:

Chamber TC GPIB:

Cold TC2 GPIB:

Hot TC1 GPIB:

Voltage V3 GPIB:

Res. V4 GPIB:

```
*RST;
:SENS:FUNC 'VOLT';
:SENS:CHAN 1;
:SENS:VOLT:CHAN1:RANG:UPP 0.1
```
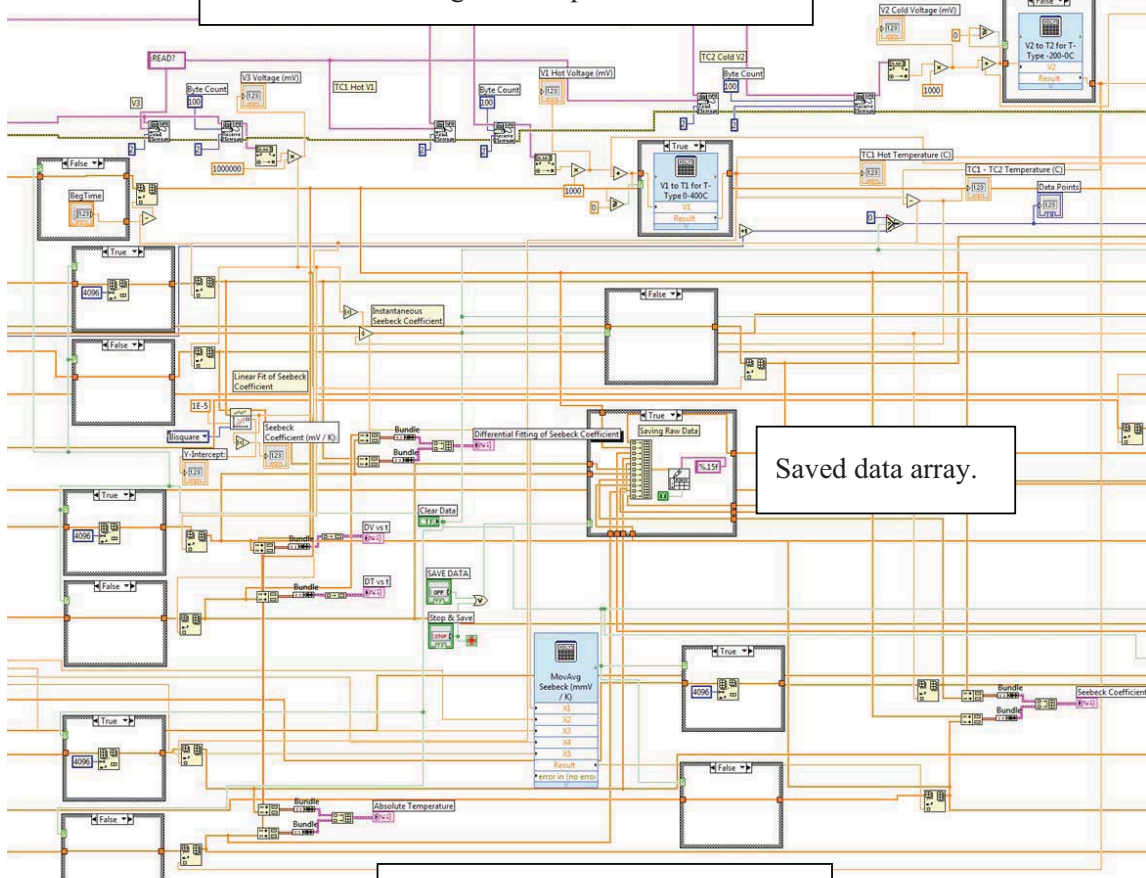
Initialization

* F3 R1 T4

BegTime

1000

Initialization of the instruments for data acquisition and the computer's clock occur outside the loop structure.
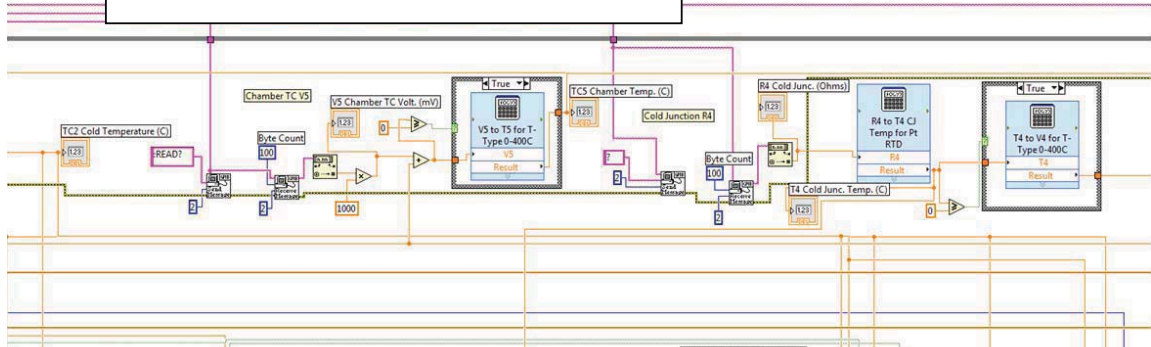
Sending and receiving data from instruments. Conversion of voltages to temperatures.
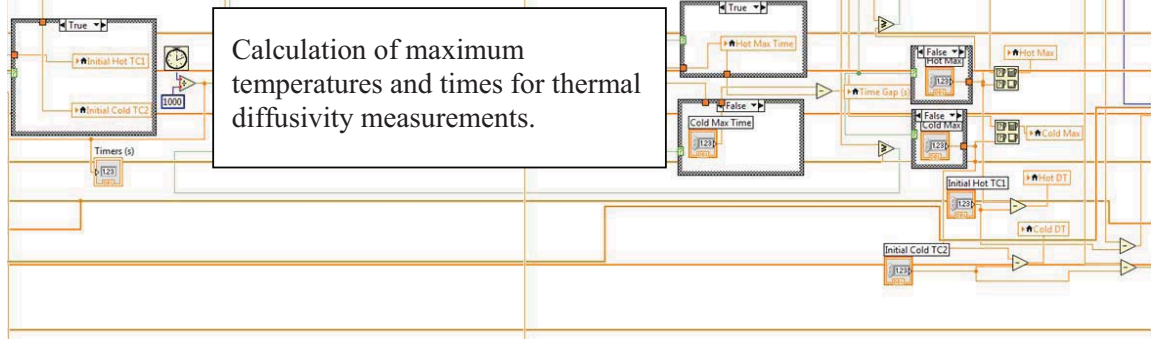
Saved data array.

Calculating Seebeck coefficients and plotting data.

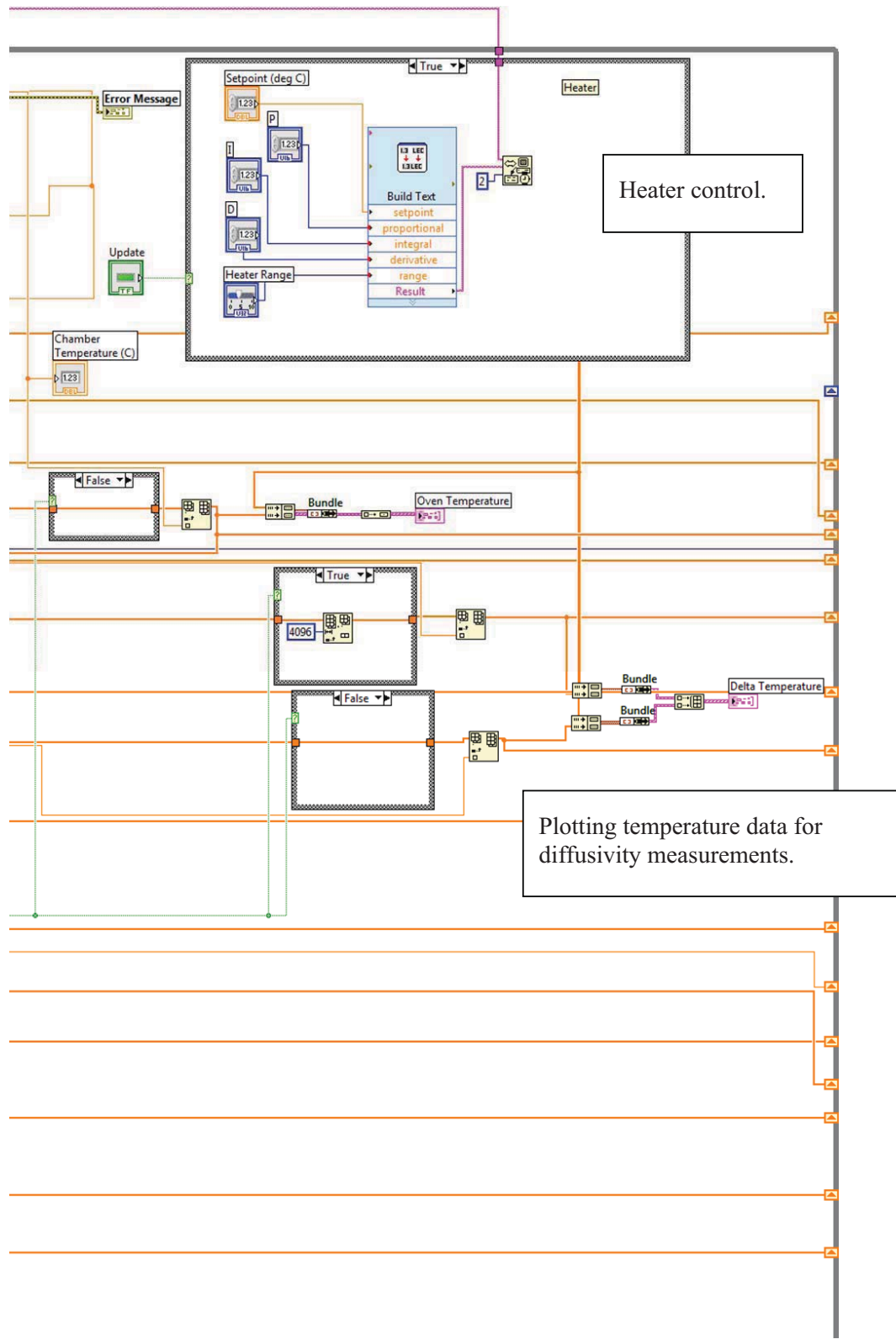Sending and receiving data from instruments. Conversion of voltages to temperatures.

Calculation of maximum temperatures and times for thermal diffusivity measurements.

Calculation of thermal diffusivity and conductivity (if applicable).

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Heater control.

Plotting temperature data for diffusivity measurements.
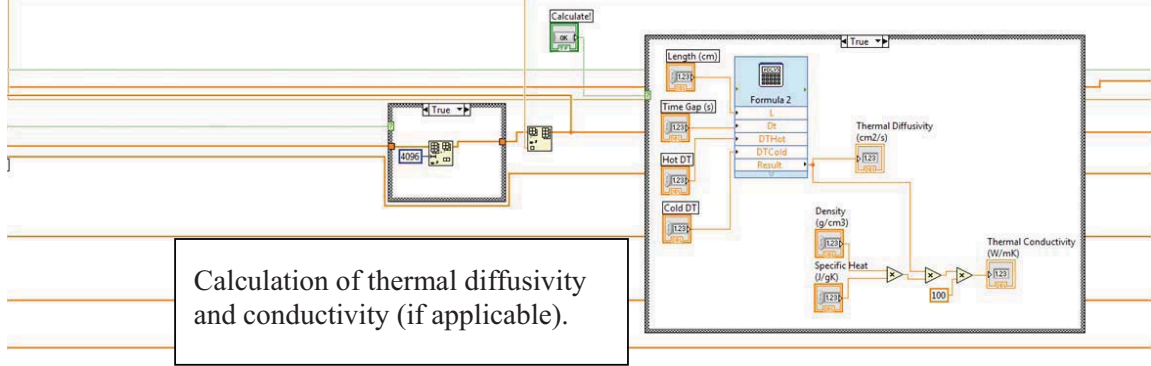
Conclusions

Thermoelectric materials are critical for direct energy conversion applications. Therefore, the development of a standard characterization system for thermoelectric research is essential for NASA and U. S. industries to accurately measure the critical parameters (*i.e.* Seebeck coefficient, thermal conductivity, and electrical conductivity) related to the performance assessment of novel thermoelectric materials.

However, there is some question as to which measurement technique(s) provides the most accurate determination of the Seebeck coefficient with elevated temperature. This has led to the implementation of nonstandardized practices that have further complicated the confirmation of reported high ZT materials. A major consideration for the procedure described was for the simultaneous measurement of Seebeck coefficient and thermal diffusivity within a temperature range. To ensure meaningful interlaboratory comparison of data, thermoelectric measurements must be reliable, accurate, and consistent.

In view of the foregoing, there is a need in the art for improved apparatuses and methods for accurately measuring the Seebeck coefficient and thermal conductivity of thermoelectric materials. These apparatuses and methods must operate at various temperatures, be simple, non-destructive, efficient, fast and affordable. There is also a need for such systems and methods to be suitable for bulk and thin film material samples in a broad range of physical types and shapes.

The thermal characterization system in this NASA-TM is designed to measure the in-plane thermal diffusivity and the Seebeck coefficient for materials in the range of temperatures from 73 K through 373 K. Custom-designed hardware and software were designed to allow for semi-automatic data acquisition and analysis. The sample holder is fabricated from stainless steel and alumina ceramic that offers high vacuum and various temperature compatibility, chemical inertness, good thermal conductivity, and electrical isolation. Two T-type thermocouple probes use tungsten wire springs to keep them in good thermal and electrical contact with the TE sample. It is these accurate and reliable measurements that allow the Seebeck coefficient and the thermal conductivity of the TE material samples to be calculated. Common sources of temperature and voltage measurement errors which may impact accurate measurement are identified and reduced. These techniques for non-destructive Seebeck coefficient and thermal conductivity measurements are simple to operate, and are suitable for bulk and thin film samples with a broad range of physical types and shapes.

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14. ABSTRACT
The Seebeck coefficient, when combined with thermal and electrical conductivity, is an essential property measurement for evaluating the potential performance of novel thermoelectric materials. However, there is some question as to which measurement technique(s) provides the most accurate determination of the Seebeck coefficient at elevated temperatures. This has led to the implementation of nonstandardized practices that have further complicated the confirmation of reported high ZT materials. The major objective of the procedure described is for the simultaneous measurement of the Seebeck coefficient and thermal diffusivity within a given temperature range. These thermoelectric measurements must be precise, accurate, and reproducible to ensure meaningful interlaboratory comparison of data. The custom-built thermal characterization system described in this report is specifically designed to measure the in-plane thermal diffusivity, and the Seebeck coefficient for materials in the ranging from 73 K through 373 K.

15. SUBJECT TERMS

Seebeck coefficient; Thermal conductivity; Thermoelectric characterization system; Thermoelectric materials

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