INL/EXT-08-14862

Measurement of Thermal Diffusivity Using a Laser Flash Thermal Property Analyzer

J. E. Daw

November 2008

The INL is a U.S. Department of Energy National Laboratory
operated by Battelle Energy Alliance

INL/EXT-08-14862

Measurement of Thermal Diffusivity Using a Laser Flash Thermal Property Analyzer

J. E. Daw

November 2008

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy
Through the INL LDRD Program
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

CONTENTS

FIGURES

TABLES

1. Introduction

This document describes the process used at the Idaho National Laboratory's (INL) High Temperature Test Laboratory (HTTL) for measuring thermal diffusivity using a Laser Flash Thermal Property Analyzer. The document is divided into three sections: Background, in which the technique is described; Setup, in which the physical system is described; and Procedure, in which the testing steps are listed and detailed; including Example Tests, in which typical tests are outlined.

2. Background

Thermal diffusivity is a material property that describes the rate at which a material will come to thermal equilibrium with its surroundings (the speed of heat propagation during thermal transients). Thermal diffusivity, denoted α, is the ratio of thermal conductivity to volumetric heat capacity:

$$\alpha = \frac{k}{\rho c_p} \tag{1}$$

Where:

α = thermal diffusivity

k = thermal conductivity

ρ = material density

c_p = specific heat capacity

The flash measurement method, shown in Figure 1, exposes one face of a thin, uniform sample to an instantaneous energy pulse. The thermal diffusivity can be calculated from the maximum temperature the opposite face reaches and the time required to reach half the value of this temperature or the 'half max time,' also shown in Figure 1. Note, curve A represents an ideal case, with no energy losses, while curves B and C represent 'real world' cases where energy losses are present and corrected for via software.

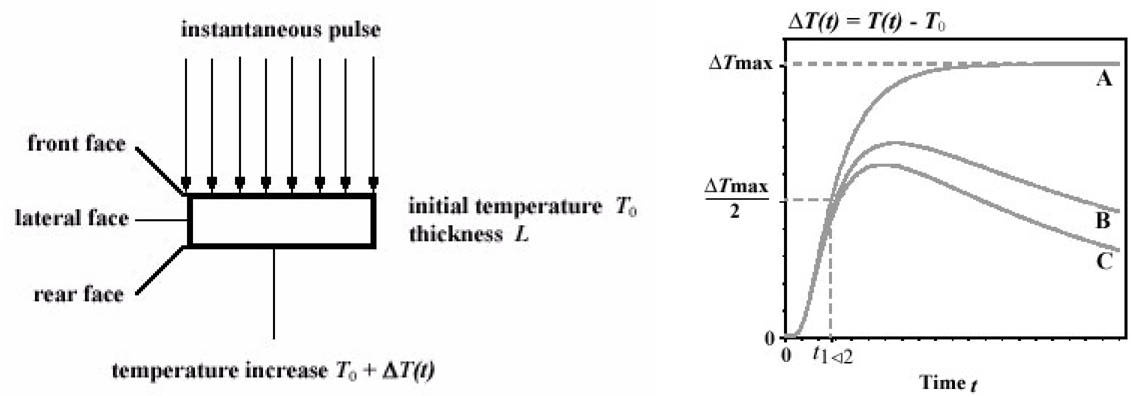


Figure 1. Diagram of flash method for diffusivity measurement.

The flash method invokes certain ideal assumptions that are unattainable in practical application. For example, the energy pulse duration may be very short, but is not instantaneous. Also, sample thickness and surface condition are not truly uniform. Therefore, a flash measurement system typically applies correction algorithms to improve the quality of results.

The ideal test case (instantaneous energy pulse, no heat losses, uniform material) was solved by Parker. Due to the non-ideal nature of real world testing, several correction algorithms are included in the FlashLine 2006 software package. Each algorithm is applied automatically during testing and may be displayed during analysis by opening the various results files that are produced. The default correction uses Clark and Taylor’s analysis. This and other schemes are described below.

- Parker: This is the ideal case solution. The thermal diffusivity is given by the relation:

$$\alpha = \frac{0.1388 \cdot L^2}{t_{1/2}} \tag{2}$$

Where:

α = thermal diffusivity

L = sample thickness

$t_{1/2}$ = time to reach half of the maximum temperature rise on the back surface

- Cowan: A correction which accounts for radiative heat losses from the sample. This correction also considers the energy pulse to be negligibly short but non-instantaneous. Thermal diffusivity is given for the Cowan analysis by:

$$\alpha = \frac{C \cdot L^2}{t_{1/2.5}} \tag{3}$$

Where:

C = a parameter related to the ratio of temperatures measured in an ideal system and temperatures measured while accounting for radiative heat losses

$t_{1/2.5}$ = five times the half maximum temperature rise time

- Clark and Taylor: The default method displayed in the FlashLine 2006 program. This technique furthers the Cowan method by deriving values of the “C” parameter at different points along the temperature rise curve.
- Heckman: The Heckman model treats, and corrects for, the energy pulse as a non-negligible triangular wave instead of a negligibly short rectangular pulse.

- Koski: An improved method in which laser pulse width corrections are incorporated into the previous methods. Also accounts for heat loss from the sample along the sample length through the use of a heat loss parameter L, given as:

$$L = [(4)(\sigma)(\varepsilon)(T^3)(D))]/k \tag{4}$$

Where:

σ = the Stephan-Boltzman constant

ε = emissivity of sample

T = average sample temperature

D= sample thickness

k = thermal conductivity

- Other’s: Several other correction factors are included which are refinements of the above methods.

The best correction to use may vary from test to test and should be verified using a goodness of fit test. This test allows the comparison of the measured (experimental) and ideal (theoretical) data. The test is performed using the equation:

$$r^2 = 1 - \frac{\sum_{i=1}^{n} (y_i - \hat{y}_i)^2}{\sum_{i=1}^{n} (y_i - \bar{y})^2} \tag{5}$$

Where:

r^2 = goodness of fit criterion

y_i = experimental data at times t_i

$\hat{y}_i$ = theoretical data at times t_i

$\bar{y}$ = mean value of experimental data in considered time interval

n = number of data points

The maximum value of r^2 is 1, this corresponds to a perfect match between theoretical and experimental data. The correction algorithm with r^2 value closest to 1 is best.

3. Setup

Figure 2 shows the Anter LaserFlash 5000 Thermal Diffusivity System installed at Idaho National Laboratory's (INL) High Temperature Test Laboratory (HTTL). The system consists of two test furnaces (referred to as tungsten and alumina furnaces), a CPU, and a laser power source (behind the alumina furnace in the figure) with a fiber optic delivery system. Software included with the system allows for control of sample holder position, furnace temperature, and test parameters as well as data recording and analysis.

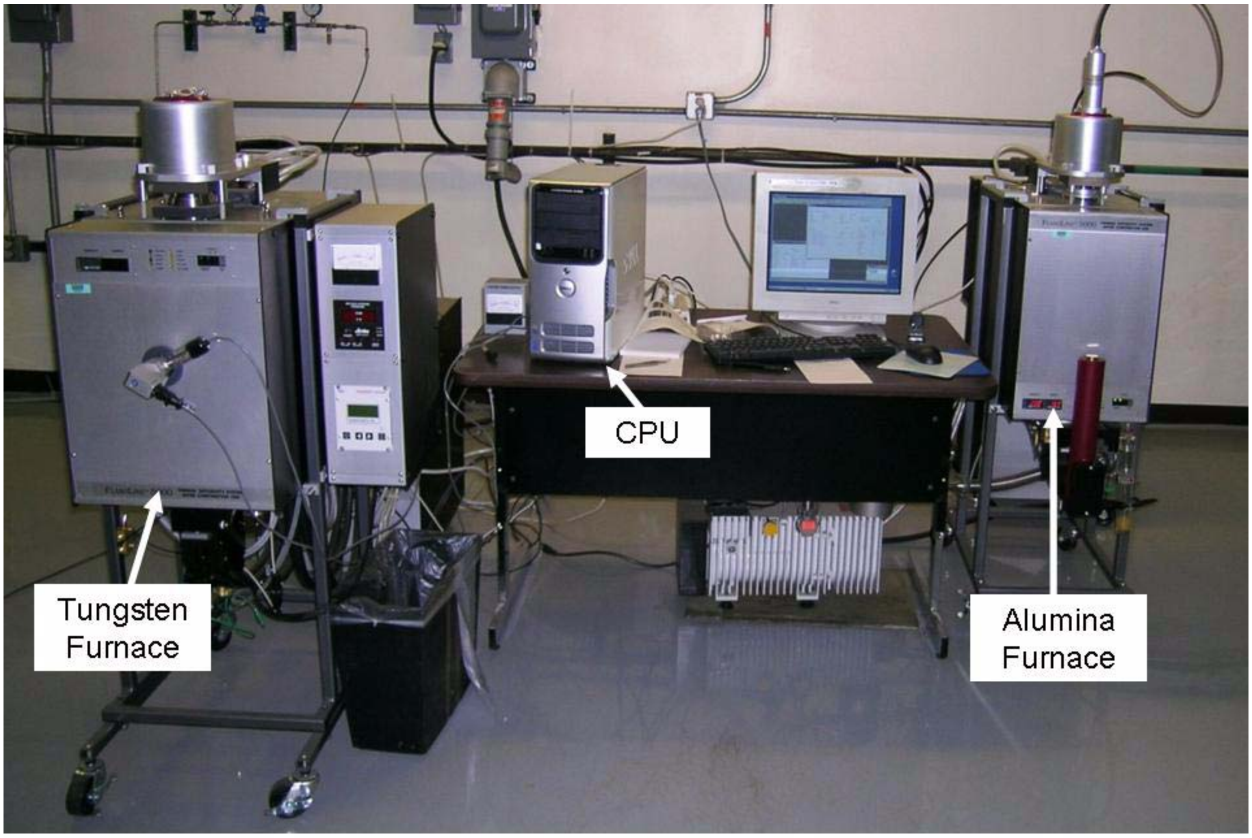


Figure 2. Anter FlashLine 5000 Thermal Diffusivity System with two furnaces.

The HTTL Anter system utilizes a separate laser power source that can easily be connected to either of the furnaces using a fiber optic wand. Figure 3 shows the laser power source as well as details of the alumina furnace. The alumina furnace generates heat (up to 1400 ^{O}C)using molybdenum disilicide heating elements. Furnace and sample temperature are measured by type-S (platinum/rhodium) thermocouples. The heat pulse generated by the laser is sensed by an indium/antimony detector (seen at the base of the liquid nitrogen dewar in Figure 3) which must be cooled constantly during testing.

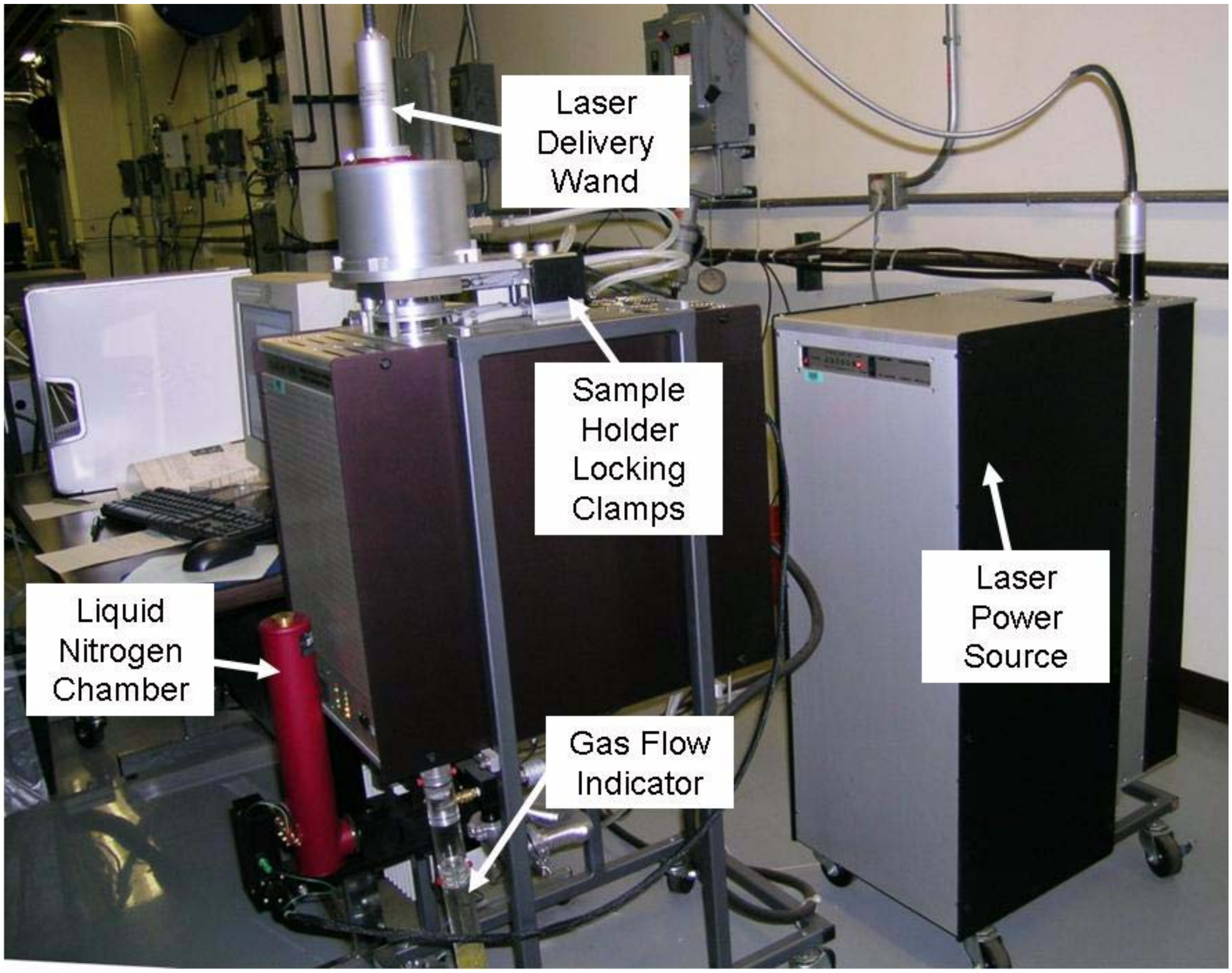


Figure 3. Laser power source and alumina furnace details.

The tungsten furnace (Figure 4) is used for higher temperature testing (up to 2800 ^{o}C), and uses tungsten heating elements with molybdenum heat shields. Furnace and sample temperatures are detected using thermocouples at lower temperatures and an optical pyrometer for high temperatures. The tungsten furnace also requires the use of a turbo vacuum pump, with tests performed in high vacuum (pressures as low as 10^{-6} torr). The two furnaces are otherwise similar.

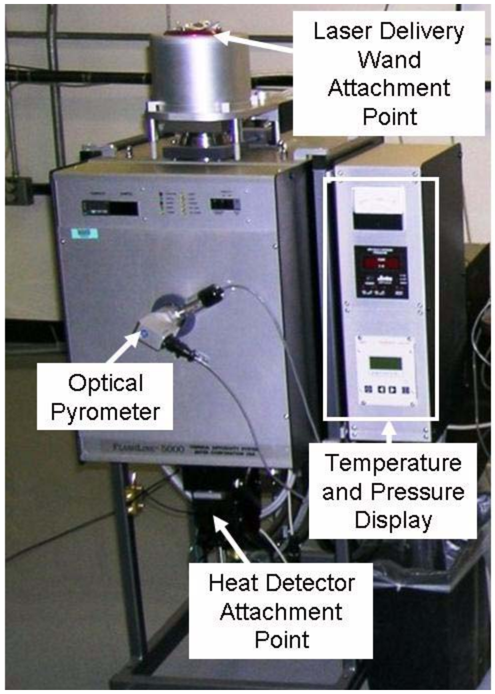


Figure 4. Tungsten furnace details.

4. Procedure

The following sections detail the testing procedure. Section 4.1 describes the general procedure used for a thermal diffusivity test. Section 4.1.1 describes the steps used for a thermal diffusivity test and describes some of the common correction techniques included in the software package. Section 4.1.2 describes the steps used for a specific heat test and details difference between a specific heat test and a thermal diffusivity test.

4.1. General Testing Procedure

Table 1 details the test procedure for this system. The test procedure makes reference to the sample holder and the loading sequence. The method for opening the sample holder is shown in Figure 5. The sample holder, its rotation during testing, and the sample loading numbering sequence are shown in Figure 6. Note, this procedure assumes that the laser wand is connected to the proper furnace.

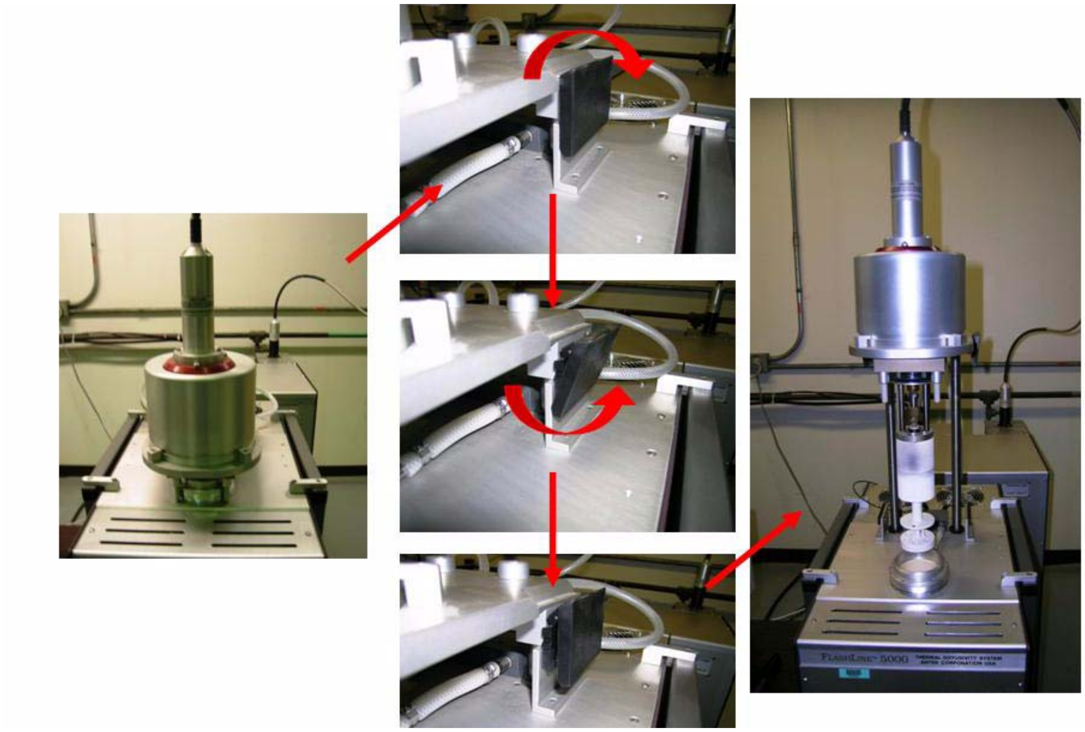


Figure 5. Sequence for opening the sample holder.

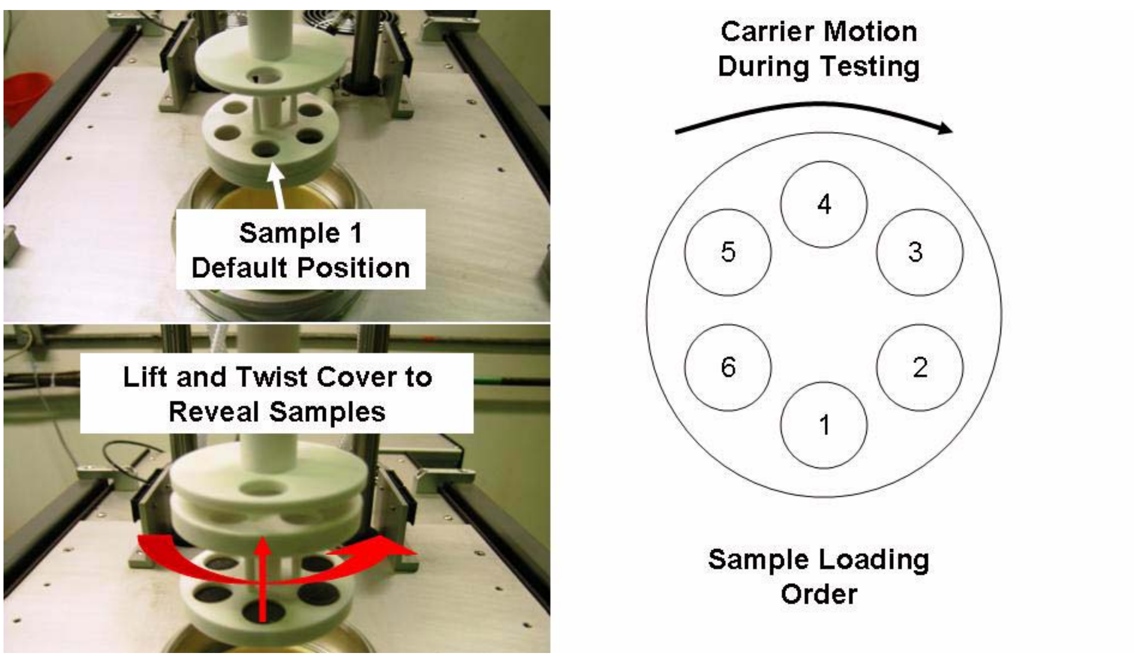


Figure 6. Sample holder characteristics including numbering sequence, reference position, and method for accessing samples.

Table 1. Test Procedure.

	Step	Action	Process	Purpose	Comments
Prepare System	**0**	Connect Furnace For Use	Connect Heat Detector, Computer And Laser Source Cables, Etc.		Not Required If Desired Furnace Is Connected
	1	Start Coolant Water Flow	Open Appropriate Flow Valve	Start Coolant Flow To Furnace	Test Will Not Run Without Coolant Water Flow
	2	Start Argon Flow	Open Tank Valve And Check Bubble Indicator On Furnace (Figure 3)	Purge Sample Holder Of Oxidizing Gases	Discrete Bubbles Should Be Present, A Continuous Jet Indicates Flow Rate Is Excessive. Pressure Regulator Should Be Set At 1-2 psi, With Fine Adjustment Done On Flow Adjustment Stem. (Note: Bubbles Will Not Be Present Unless Furnace Is Closed)
	3	Start CPU	Press On/Off Switch on Computer And Log On	Allows Test Programming, Data Logging And Processing	
	4	Start Laser Power Source	Set Toggle Switch On Front Of Laser Source To On	Provide Laser Pulse For Testing	
	5	Start Furnace Power Source	Set Main Power Switch On Back Of Furnace To On	Start Power To Furnace	
	6	Fill Liquid Nitrogen Container	Lift Cap Off Container and Move Nitrogen From Dewar To Container Using Small Flask	Cool InSb Heat Detection System	Proper PPE Must Be Used. This Includes Gloves, Lab Coat, Safety Glasses, Safety Shoes, And Face Shield

Table 1. Test Procedure.

Prepare Sample	**7**	Clean Samples	Using Proper PPE, Thoroughly Clean Samples With Acetone	Eliminate Impurities Which Could Alter Sample Thickness Or Emissivity	The Sample Should Be Handled With Gloves Or Tweezers from This Point On
	8	Measure Samples	Measure Samples With Micrometer. Take Average Of Three Measurements	Get Average Thickness Measurement	Accurate Thickness Measurement Is Necessary For Program (Samples Should Be 1-6 mm Thick With Faces Parallel To Within 0.5% Of The Thickness.)
	9	Open Sample Holder	Unlock Clamps On Sides Of Laser Wand Mount (Figure 5)	Allow Access For Loading Samples	Once Clamps Are Unlocked Sample Holder Rises Automatically
	10	Load Samples	Lift And Twist Sample Cover (Figure 6), Always Have Six Samples In Sample Holder	Allow Measurement Of Sample Diffusivity	Numbering Is Counterclockwise. Not All Sample Positions Need To Be Tested, But Do Not Leave Empty Positions
	11	Secure Samples	Rotate Sample Cover and Lower Onto Sample Holder	Secure Samples While Sampler Holder Moves	Do Not Leave Sample Cover In Up Position, It Will Block Laser Pulse
	12	Close Sample Holder	Lower Sample Holder And Re-Lock Clamps	Position Sample Holder In Furnace	
	13	Vacuum Purge Furnace	Activate Vacuum Pump And Alternate Vacuum And Purge Gas With Valving	Remove Oxygen From Furnace	Oxygen Present In The Furnace Could React With Samples, Altering Their Thermophysical Properties (Note: Tungsten Furnace Requires Roughing And Turbo Pump Use)
Prepare Test	**14**	Open FlashLine 2006 Program	Double Click On Icon	Start Measurement And Control Program	
	15	Begin Programming Test	Under Operation, Select Startup Information	Start Interactive Programming	A Series Of Pop-Up Screens Will Guide The User Through The Programming Process
	16	Program Test Steps	Follow On-Screen Prompts		Steps Are Explained More Thoroughly In An Example Test In The Next Section
	17	Activate Hardware And Interlock	Press Appropriate Buttons On Furnace Panel When Prompted		Test Will Not Run Until Hardware And Interlock Are Activated
	18	Start Test	Click OK When Prompted And Ready		Testing Process Is Automated

Table 1. Test Procedure.

Analyze Data	**19**	View Data In FlashLine 2006	Use Plotting Buttons In FlashLine Window	View Data and Plots From Current And Previous Tests	Data, Plots, And Curve Fits May Be Viewed In The FlashLine Program Window
	20	Export Shot Data To Excel	Select Post Analysis, Shot To Text File: Sample Folder, Select Sample, Double Click on Data File	Copy File as Text File That May Be Opened In Excel	Each Sample Must Be Converted Separately, Each Shot Must Be Transferred Separately
	21	Export Diffusivity Data To Excel	In Excel: Open> C:Drive>Anter>Data>Test Number>Sample>Results File		Each Sample File Contains Many Results Files, Each Uses Different Correction Algorithms
Shut Down	**22**	Close Software	Close Program Window		
	23	Shut Down Hardware	Reverse Steps 1 Through 5		
	24	Remove Samples	Reverse Steps 9 Through 12		Only After Samples Have Cooled!

4.1.1. Representative Thermal Diffusivity Test

This section details an example test for the purpose of clarifying the testing process. Details provided in this section follow the procedure outlined in Table 1.

Step 0: Ensure that the system is connected to the desired furnace. The furnace must be connected to both the CPU (for control and data collection) and to the laser power source (for laser control and feedback). First, install the heat detector assembly by sliding the detector into position, securing the locking clamps, and connection the ground and computer connection cables. These steps are shown in Figure 7.

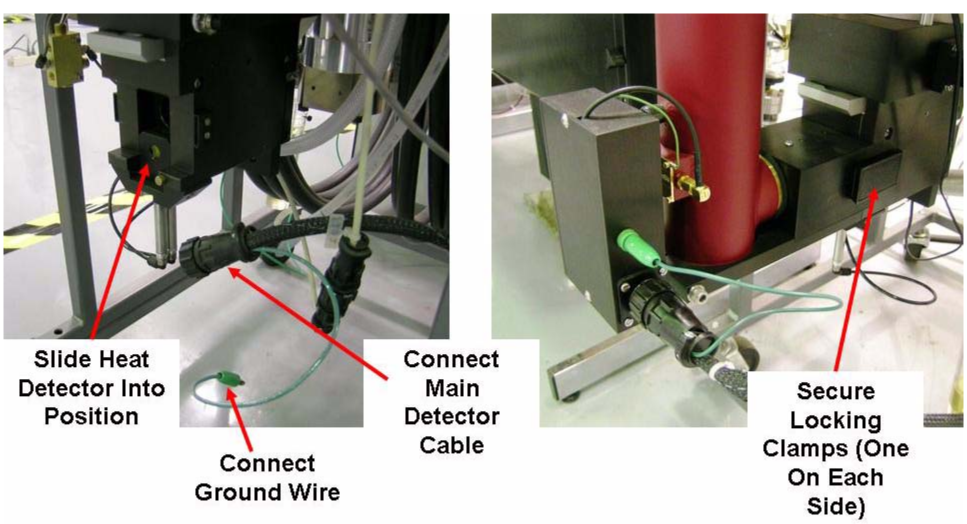


Figure 7. Heat detector installation.

Next, connect the laser source to the desired furnace. With the laser power source unplugged, disconnect the laser wand (if it is not connected to the desired furnace) and reconnect it to the desired furnace, as shown in Figure 8 (for switching from the alumina to the tungsten furnace).

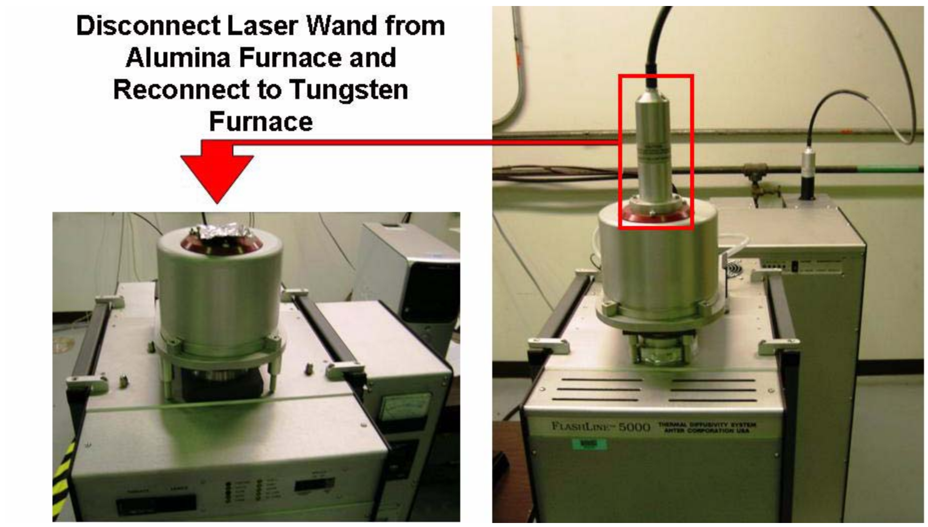


Figure 8. Laser wand installation.

The laser source is also connected to the furnace via a data cable, for controlling the laser. Figure 9 shows the data cable connection installed in the alumina furnace and the cable port on the tungsten furnace.

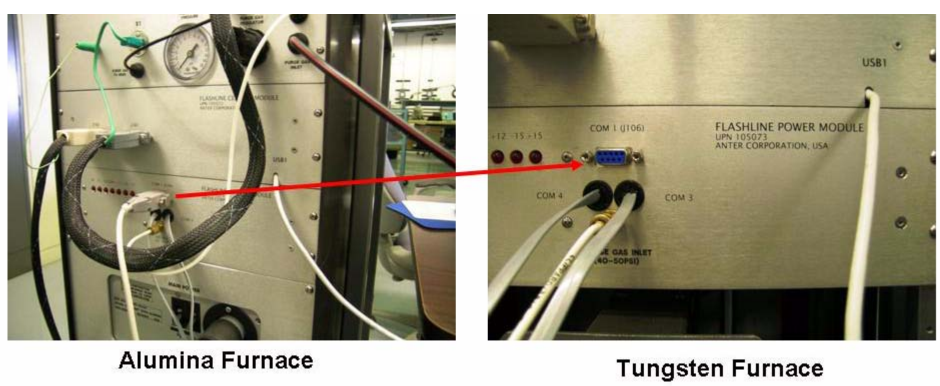


Figure 9. Laser control cord installation.

Three cables connect the furnace to the CPU. The connections are labeled on the back of the CPU casing as USB 1, A, and B. USB 1 is a USB cable, while A and B are data cables. On the back of the furnace the cables are marked as USB 1, COM 3 (corresponding to B), and COM 4 (corresponding to A). The back of the CPU, with cables labeled, is shown in Figure 10.

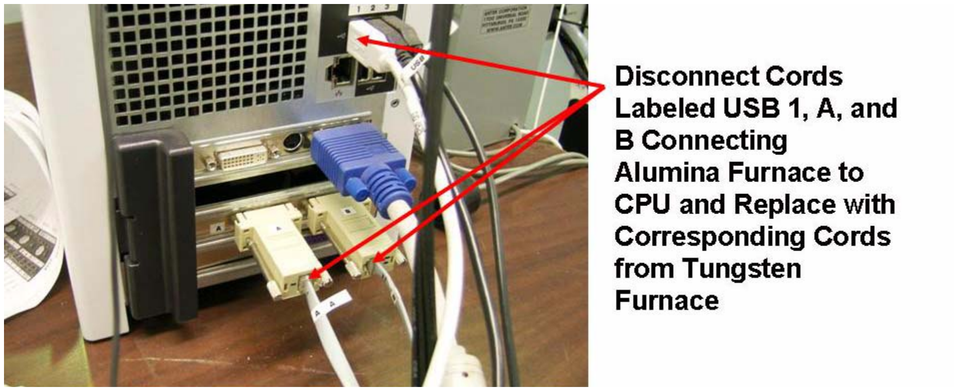


Figure 10. Cable connections on back of CPU.

Vacuum pumps are available for each of the FlashLine 5000 furnaces. If a vacuum pump-down/inert gas purge cycle is desired when using the alumina furnace, an external roughing vacuum pump must be connected via a flange located at the bottom of the furnace. The tungsten furnace must be run with samples in vacuum conditions or an inert purge gas (such as argon) to protect the heater elements. The vacuum level is dependant on the furnace temperature. The vacuum may range from 10^{-6} torr at room temperature to 10^{-3} torr at 2800 ^{o}C. Roughing and turbo-pumps must be connected to the tungsten furnace in order to achieve the required high vacuum conditions. The pump and connections are shown in Figure 11.

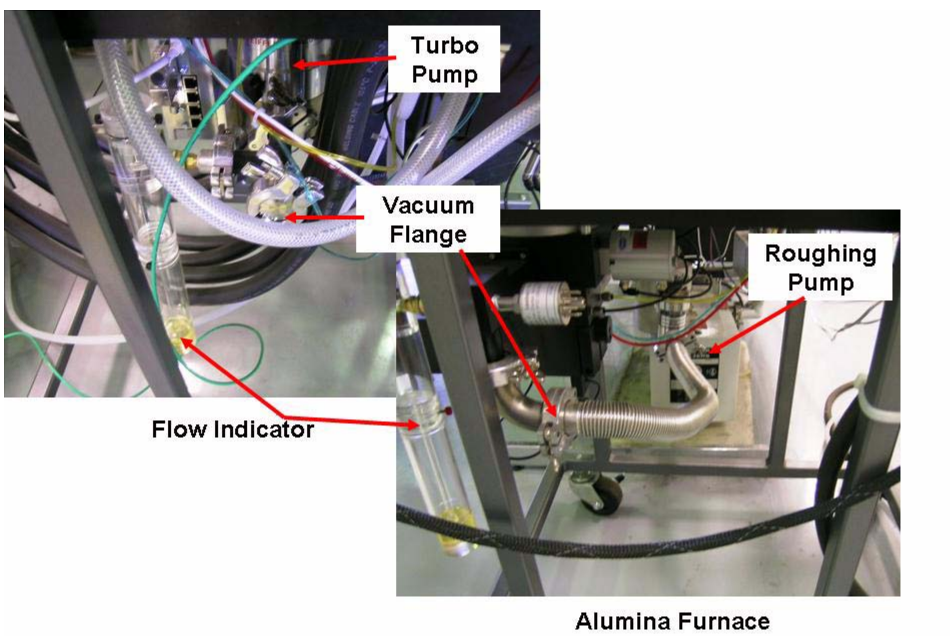


Figure 11. Vacuum Connections and Purge Gas Flow Indicators

Step 1: Start coolant water flow by turning the appropriate valve on the water supply. The water flow can be verified at the outlet.

Step 2: Start argon flow by opening the appropriate argon cylinder after making sure that the correct valves are open along the argon routing tubes. Argon flow can be verified by visually inspecting the flow indicator on the furnace. The indicator uses a bubbler, argon should create bubbles in the oil. If the argon creates a continuous jet in the oil, the flow rate is too high. The flow rate may be adjusted by using the fine pressure control, as shown in Figure 12. Adjusting the pressure regulator will also change the flow rate, but the pressure indicator should read between 1 and 2 psi (the settings at installation). Note: The tungsten furnace does not have an integrated pressure regulator.

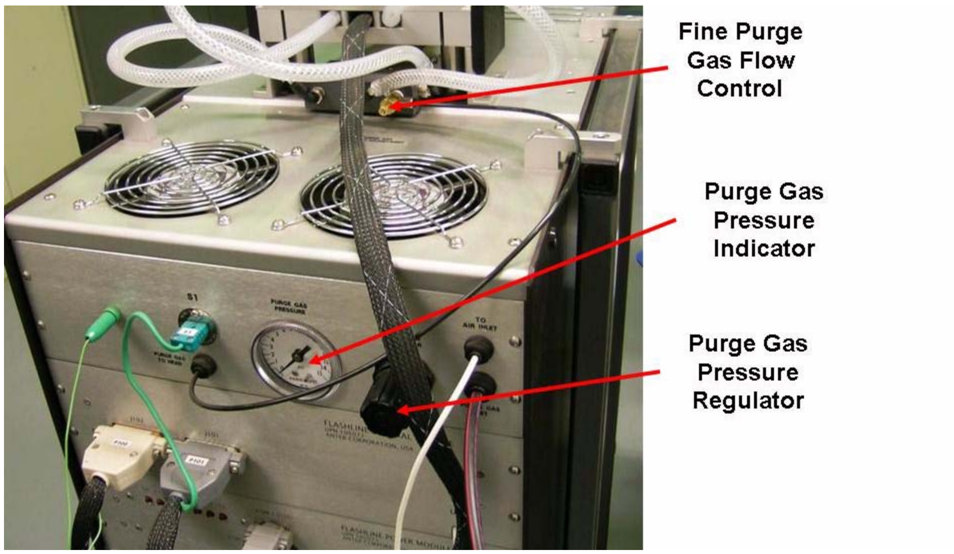


Figure 12. Purge gas flow control on the back of the alumina furnace.

Step 3: Start CPU and monitor by pressing the power buttons.

Step 4: Start laser power source by setting the toggle switch on the front of the laser power source to the on position. With the power source operating, check the coolant level and clarity. The coolant and coolant pump are located near the back of the power source, opposite the laser wand mount, in a separate enclosure from the rest of the power source components. The coolant tank and pump are visible through a gap in the casing. The coolant should be clear and fill the tank such that the stainless steel exit pipe (entering the reservoir from the top) is partially submerged (at least 1 to 2 inches). The sound emitted by the pump should be constant, not changing in pitch or amplitude. Use of the LaserFlash system should be discontinued if the pump and coolant are not operating properly, as the pump and laser could be damaged.

Step 5: Start furnace power source by setting the toggle switch on the back of the furnace power source to the on position.

Step 6: Fill liquid nitrogen container. This is accomplished by first removing the brass colored cap from the top of the container (shown in Figure 3). Position a small funnel in the hole from which the cap was removed. Wearing proper Personal Protective Equipment (leather gloves, lab coat, safety glasses, safety shoes, and a face shield), transfer liquid nitrogen from the large dewar into a smaller flask by positioning a funnel into the flask and holding the flask under the nozzle of the dewar and opening the flow valve. Care must be taken to avoid over filling the flask. Because there is no level indicator, this requires periodically stopping, removing the funnel, and visually inspecting the amount of nitrogen in it. Close the valve and pour the liquid nitrogen from the flask into a funnel positioned in the laser flash system liquid nitrogen container. Repeat the transfer process until the laser flash system liquid nitrogen container is full, which is indicated by a small amount of liquid nitrogen spilling from the top of the container. Replace the cap into the top of the container. A full container of liquid nitrogen should allow for up to eighteen hours of testing.

Step 7: Clean the sample with acetone or isopropyl alcohol. This will remove any contaminants that might react with the sample at high temperatures or change the thickness or emissivity of the sample. After this step, the sample should be handled with gloves or tweezers.

Step 8: Measure the thickness of the sample using a micrometer. The average of three random measurements *should* be used. This measurement will be entered into the computer and is used for calculation of the thermal diffusivity.

Step 9: Open the sample holder using the sequence shown in Figure 5. The clamps located on top of the furnace (behind the laser wand) must be released. The sample holder will then rise on its own (it is counter balanced).

Step 10: Lift the sample cover and twist (as seen in Figure 6) such that it sits on the vertical prongs, allowing loading of the samples. Load the samples starting in position 1. Up to six samples may be loaded at one time. It is not required to test all sample positions, but there must not be any empty positions.

Steps 11 and 12: Twist and lower the sample cover to secure the samples. Lower the sample holder and re-lock the clamps.

Step 13: If necessary, vacuum purge the alumina furnace by activating the vacuum pump and alternately opening and closing the isolation valve between the pump and furnace. This will alternate the atmosphere between vacuum and inert purge gas within the furnace. If the tungsten furnace is used, both roughing and turbo pumps are used. With the roughing pump active, the turbo-pump is manually activated using the front control panel on the furnace. The turbo pump may be activated after the roughing pump has achieved a vacuum of at least 100 torr. The turbo pump must be run until the test is complete and the furnace is at room temperature. After the turbo pump is deactivated, it will take about 15 minutes for the pump to completely stop. At this time the roughing pump may be stopped and the purge gas flow started. Only after the purge gas bubbler indicates flow may the furnace be opened.

Step 14: Open the software package by double clicking the FlashLine 2006 icon on the computer desktop.

Step 15: Under the operation heading on the toolbar, select “Startup Information.” This will initiate software that guides the user through the process of programming a test. The drop-down menu is shown in Figure 13 with the proper command highlighted.

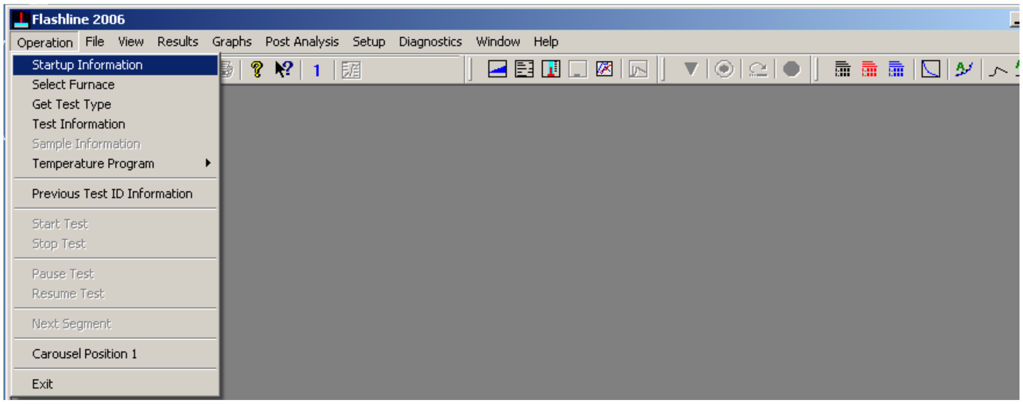


Figure 13. Operation drop down with Startup Information highlighted.

Step 16: Program the test by following the on screen prompts. The first prompt (Figure 14) requests identification of the furnace to be used. The FlashLine 5000 in use at the HTTL utilizes two furnaces. Select the desired furnace and click on “OK.” A confirmation pop-up appears, select “OK.” Another pop-up requests confirmation that the controller parameters file will be used, if so click “Yes.” Test type options are next presented. For the HTTL system the options are normal test (diffusivity) and specific heat run. To test diffusivity, select normal test and click “OK.”

Figure 14. Furnace selection prompt.

A list of temperature programs appears (Figure 15). Select the appropriate program or choose to create a new program. Any existing program may be modified, so it may be easier to select an existing program and alter it. The program allows 99 programs to be saved.

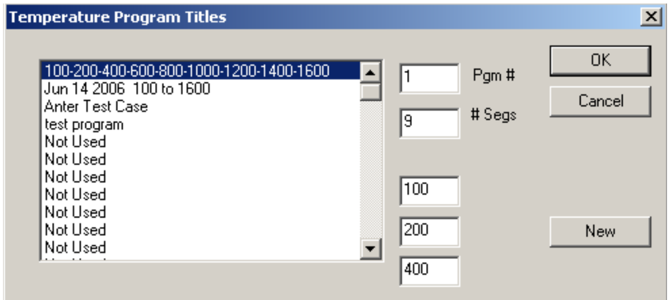


Figure 15. Temperature program selection prompt.

The screen shown in Figure 16 allows modification of the selected program. Test segments may be added or removed, and all test parameters may be changed. The buttons labeled uniform rate, uniform shots, and voltages allow the same value to be assigned to each segment. The uniform temps button allows the user to enter a starting and end temperature and a uniform temperature increment. When the program is ready, select "OK." Ramp rates should be determined based on materials being tested. The rate must be slow enough to avoid thermal shock (which could cause cracking of samples). Material property data should be researched, when available, prior to testing. The number of shots taken should be chosen such that the test will preclude excessively long tests (as liquid nitrogen may run out after about 16 hours) but allow for an average of good data to be acquired. The vendor recommended default value is 3 shots per sample per temperature. Laser voltage is automatically varied by the system. The default start value is 1600 V, but the system will adjust this as necessary for optimum performance.

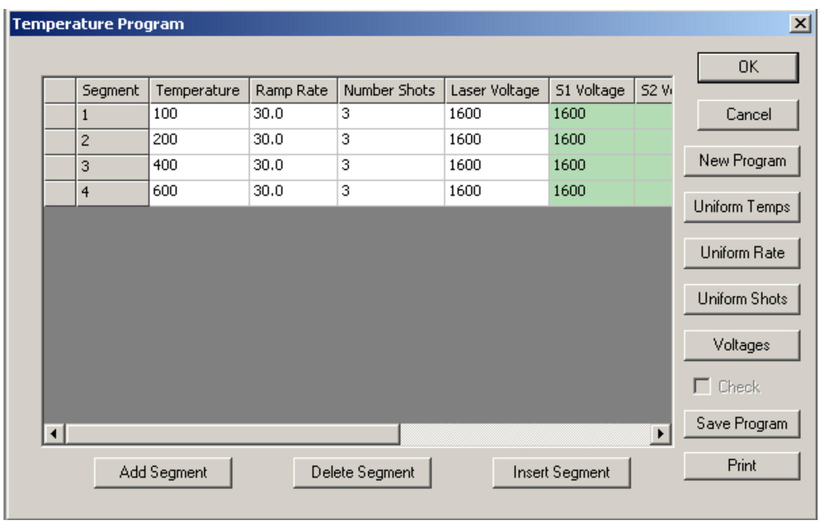


Figure 16. Temperature program modification screen.

The screen shown in Figure 17 allows for recording of test identification information. Enter operator ID and test title. The test number automatically increments but may be changed. Click "OK."

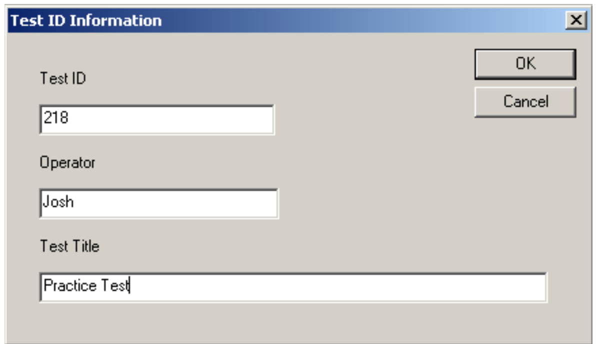


Figure 17. Test identification information screen.

Sample data are entered in the window shown in Figure 18. The sample holder for the alumina furnace allows testing of up to six samples in a single test. Once all information for a sample is entered click “Another” to enter data for the next sample. Once data are entered for all samples, click “OK.”

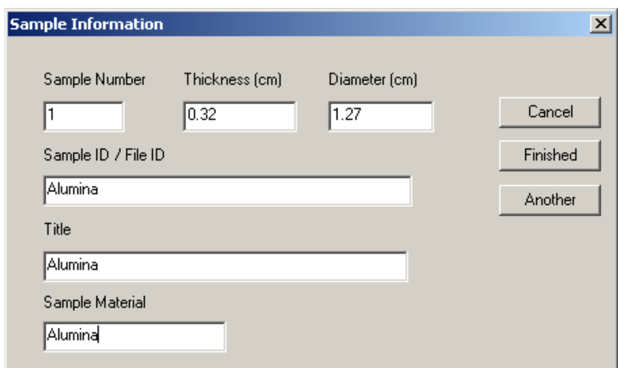


Figure 18. Sample information screen.

The screen shown in Figure 19 prompts the user to enter a safety temperature. Typically, 50 °C above the maximum test temperature is used. The furnace may heat 20-30 °C above the set temperature in order to bring the sample up to temperature. The furnace will shut down, and an alarm will sound if the safety temperature is reached. Click “OK.”

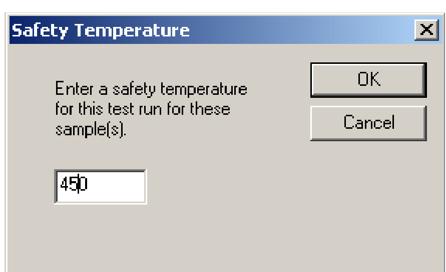


Figure 19. Safety temperature setting prompt.

The next prompt, shown in Figure 20, will request data acquisition settings for each sample. Laser power level, data acquisition rate and data acquisition time may be altered. Acquisition rate and time should be varied according to the expected thermal diffusivity of each sample. A sample with lower thermal diffusivity will have a longer half max time (see Section 2). Therefore the sampling time must be increased to compensate or part of the heating curve will be omitted from the analysis. If the sampling time is increased, the sampling rate should be decreased proportionately to avoid making data files too large. When the settings are correct click "OK," this must be done for each sample. A pop up will ask "Do you wish to start the test now?" If the settings are correct, click "Yes."

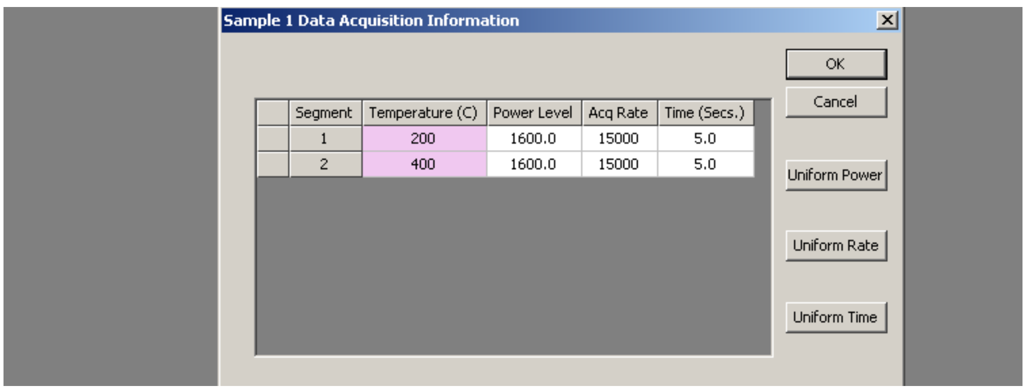


Figure 20. Data acquisition information screen.

Step 17: Press the enable hardware button on the front control panel on the furnace when the screen shown in Figure 21 appears. Press the button until the green light appears. Pressing this button before this screen apprears will have no effect. Once the light is on click "OK." A warning to make sure all hardware is on will appear, check and click "OK.". A warning to make sure that coolant water, purge gas, vacuum, and liquid nitrogen are ready next appears. Check water, gas, and nitrogen and click "OK."

Figure 21. Hardware enable prompt.

When the screen shown in Figure 22 appears press the manual interlock button on the furnace front control panel and click “OK.”

Figure 22. Manual interlock enable prompt.

Step 18: If all steps above have been completed and the program is set as desired, press “OK.” when the start test prompt appears. The test will run automatically. The screen shown in Figure 23 shows some test windows that give information in real time.

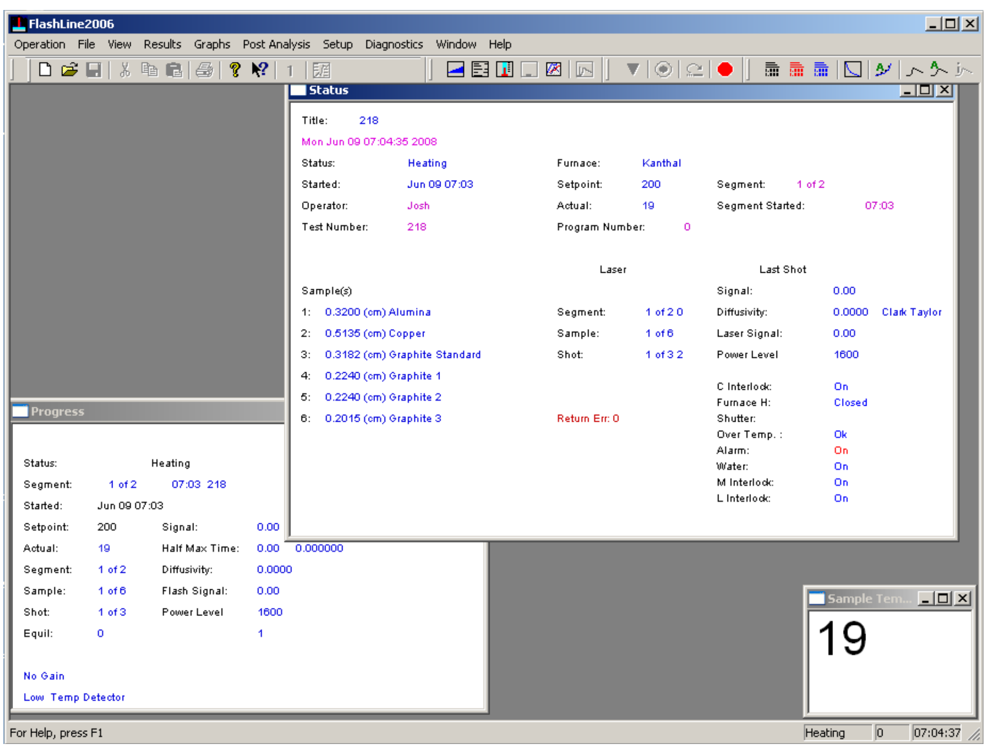


Figure 23. Test status windows.

The windows can be selected from the tool bar at the top of the screen. When the laser is activated for a test shot, the window shown in Figure 24 will automatically appear. This window shows data collected from the test shot, including laser power (red line) and heat detector output (black line) as functions of time.

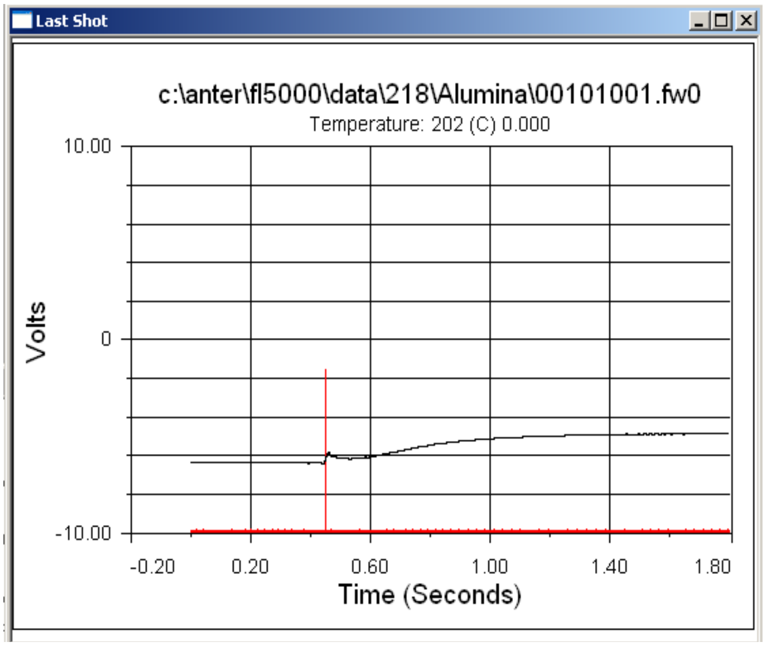


Figure 24. Last shot data window.

Step 19: To view test data in the FlashLine 2006 software, use the buttons highlighted in the red box in Figure 25.

The functions, from left to right, are:

- Text Diffusivity Results: This option displays diffusivity results in a tabulated format.
- Text C_p Results: This option displays specific heat capacity results in a tabulated format.
- Text Conductivity Results: This option displays thermal conductivity results in a tabulated format.
- Graph Results: This option plots the data reported in the text result options.
- Curve Fit Text: This option reports the same information as the text results options, but allows the use of either a polynomial or spline curve fit.
- Shot Thermogram: This option displays the laser pulse and optical detector output for a single test shot.
- Shot CDV: This option displays all shot data in a text format.
- Shot Info: This option displays all diffusivity values for a sample.

Figure 25. FlashLine 5000 Results Options

Step 20: To export shot data (laser pulse voltage and heat detector voltage as functions of time) to a spreadsheet program for processing, select the "Post Analysis" option on the toolbar as highlighted in Figure 26. The "Shot to Text File" option will convert the recorded data for one laser shot to a text file. Using this option, each shot must be converted separately. The "Shot to Text File Sample Folder" option (highlighted in Figure 26) will convert all shots for a single sample simultaneously. Using this option, each sample must be converted separately. Figure 27 shows a single shot file highlighted. Double clicking the file will convert the file if "Shot to Text File" is used. If "Shot to Text File Sample Folder" is used, double clicking the selected file will convert all files in the folder.

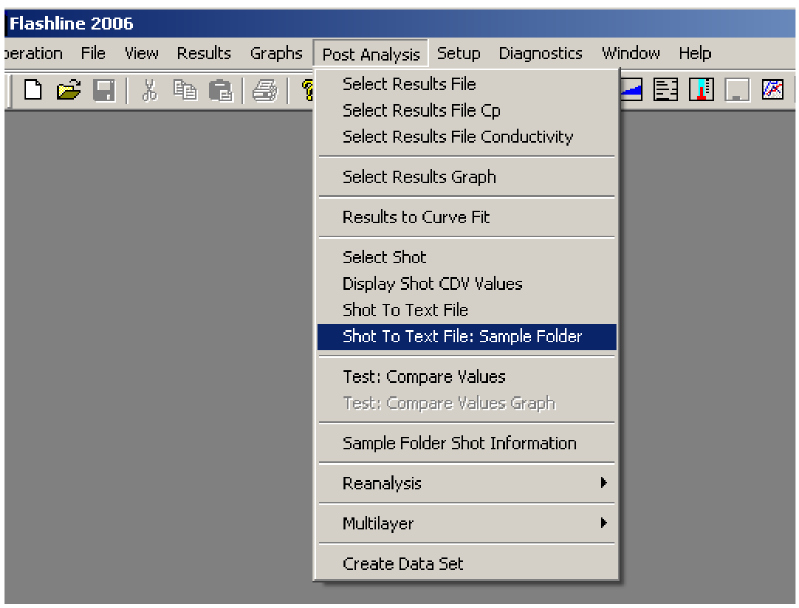


Figure 26. Shot data file to text file conversion option.

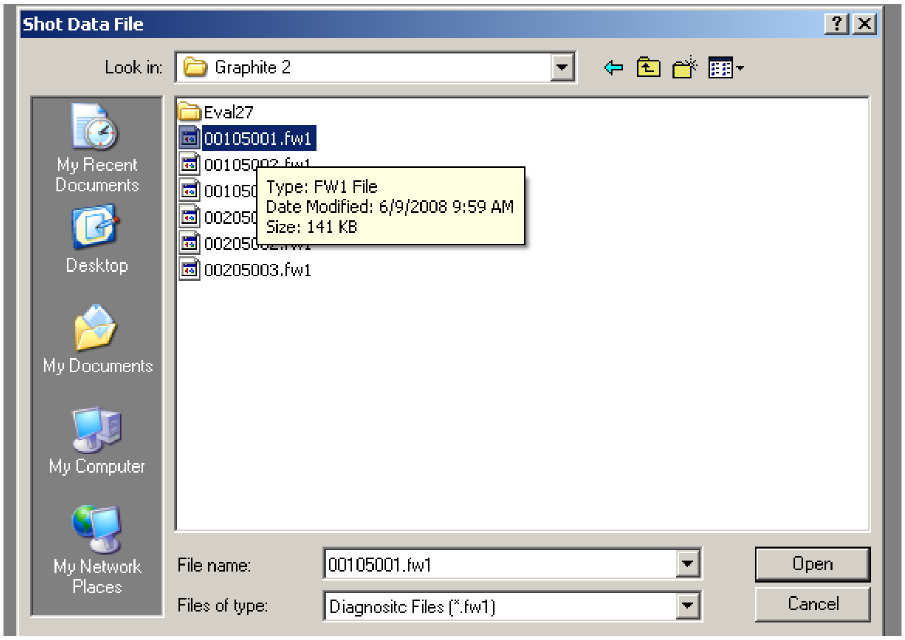


Figure 27. Shot data file selection.

Step 21: Windows explorer is the best option to export test results to a spreadsheet program. From the windows desktop, press the start button. Under Accessories, select Windows Explorer. Choose the drive corresponding to the main hard disc drive for the computer (C: in the HTTL system). Click the “Anter” sub-folder. Open the “FL5000” folder and click on “Data.” Choose the folder corresponding to the test number desired. The sub-folders within are named after the test samples (entered during the test programming phase). Select the sample of interest. The files named results may all be opened in a spreadsheet program. The file extensions correspond to the type of correction algorithm that was used on the raw data to compute the thermal diffusivity. The file hierarchy is shown in Figure 28.

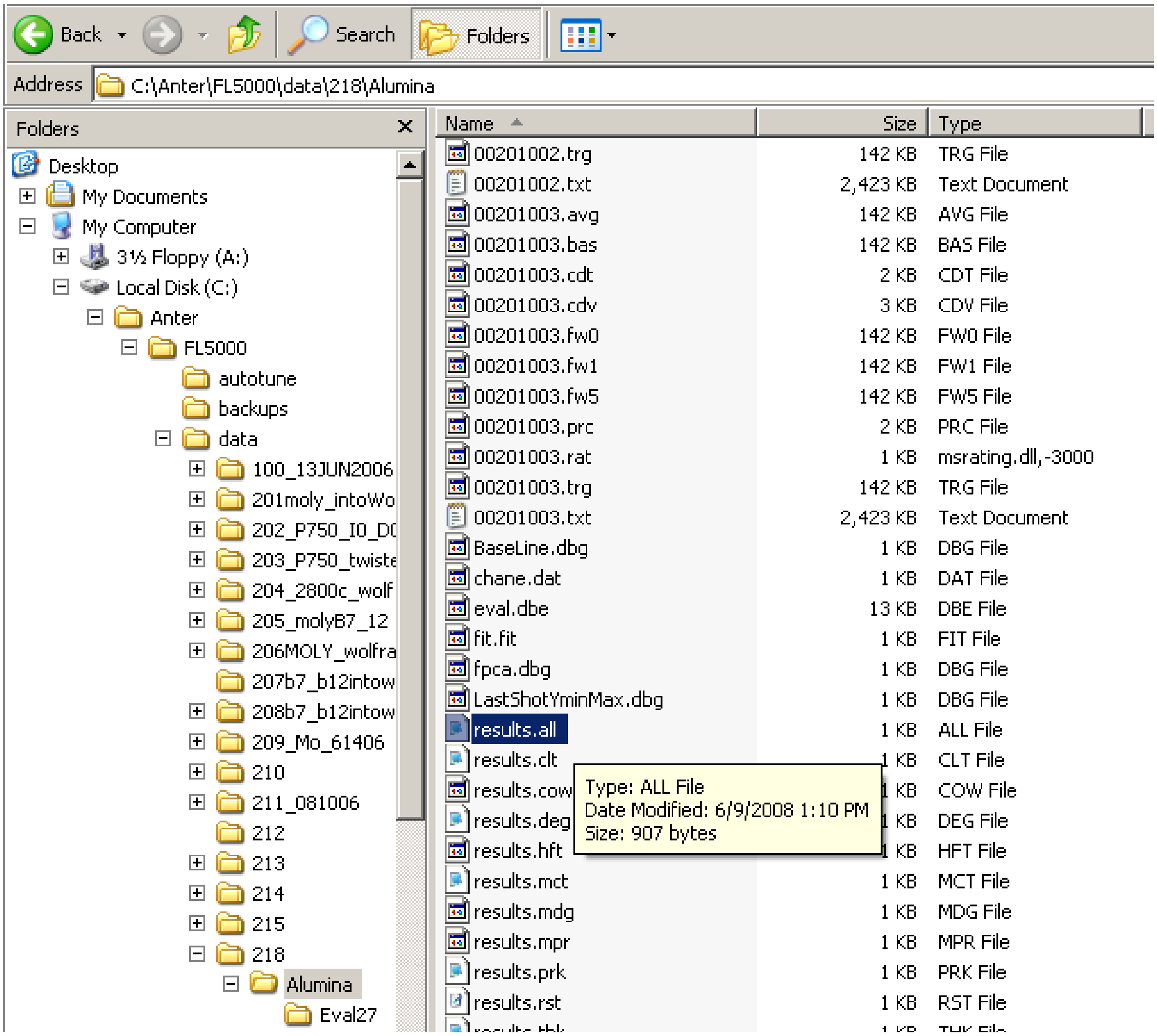


Figure 28. Results file hierarchy.

File named results can be compared to determine which correction method works best for a specific test. Files with ".fw0" extensions are shot data. Laser power and heat detector output are recorded as functions of time. These files may be converted, using the software, to text files and used with spreadsheet programs. Other files in the sample folders are used by Anter for diagnostic purposes.

Step 22: After the tests have finished and all data processing and export are complete, shut down the software by closing the program window.

Step 23: Shut down the system hardware by performing steps 1 through 5 in reverse order and turning systems off instead of on. This step should only be performed after tests have finished and the system has cooled to a safe level (the program automatically cools to 50 ^{o}C). Recall that, if the tungsten furnace is used, the turbo pump must be run until the test is complete and the furnace is at room temperature. After the turbo pump is deactivated, it will take about 15 minutes for the pump to completely stop. At this time the roughing pump may be stopped and the purge gas flow started. Only after the purge gas bubbler indicates flow may the furnace be opened.

Step 24: Repeat steps 9 through 12, this time removing the samples. This step must only be performed after the samples have cooled completely.

4.1.2. Example Specific Heat Test

The procedure for testing specific heat is the same as for thermal diffusivity, with a few minor variations. Changes to procedural steps are as follows (other steps are unchanged):

Step 10: To measure the specific heat capacity of a sample, a reference must be used. The reference sample must be loaded in carrier position 1, as shown in Figure 6.

Step 16: Select "Specific Heat Run" instead of "Normal Test." When entering sample data, further prompts will ask for density file data. For the reference sample (provided by the manufacturer), select the appropriate file from the display window. For experimental samples reference files must be created using ReferenceMaker, a separate software program provided by the manufacturer. Steps for creating a reference file are detailed in the next section.

The remaining steps are unchanged from the thermal diffusivity test.

4.1.2.1 Creating a Density Reference File

The ReferenceMaker software package allows creation of a reference file from known material property data. This file may be used in conjunction with the FlashLine 5000 software to measure the specific heat capacity of experimental samples (density files are needed for specific heat measurements). Reference files must be created for each experimental sample.

Step 1: From the computer desktop, open the RefMaker.EXE file.

Step 2: Select "Operation"=>"Create Reference File."

Step 3: Select "Density" from the pop up menu (shown in Figure 29), and click "OK."

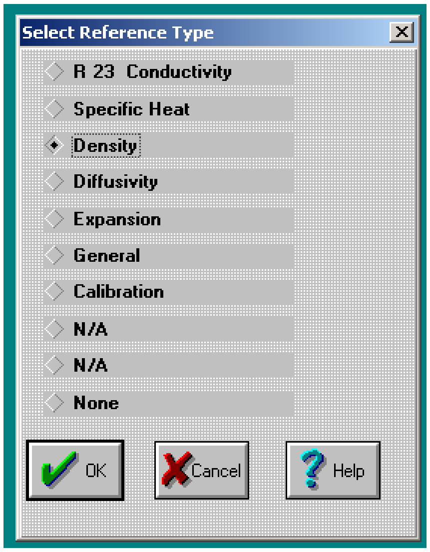


Figure 29. ReferenceMaker reference type selection menu.

Step 4: Enter sample identification information in the pop-up screen shown in Figure 30. This information should correspond to the sample to be tested.

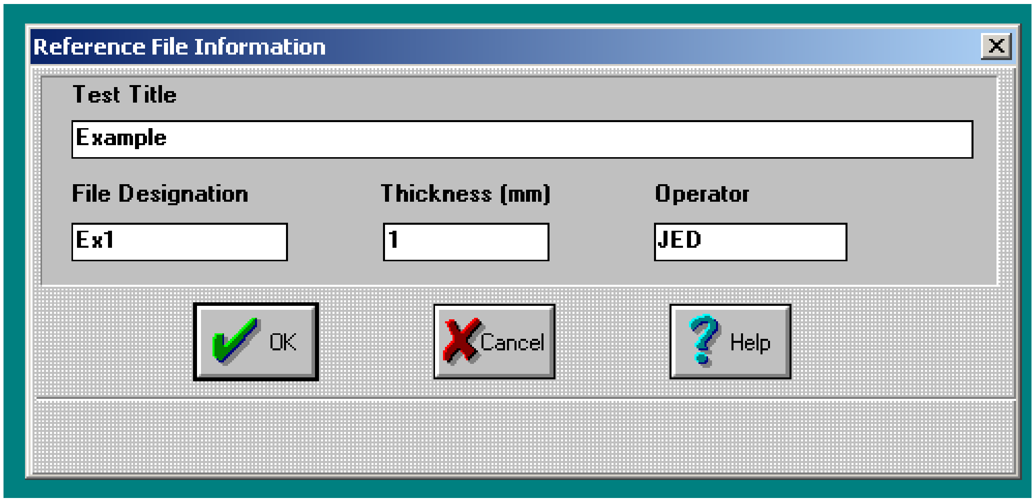


Figure 30. Reference file sample identification.

Step 5: Enter temperatures and corresponding sample density data in the table shown in Figure 31. A pop-up warning will appear stating that the R23 parameters have changed. Click "Yes" to accept the changes.

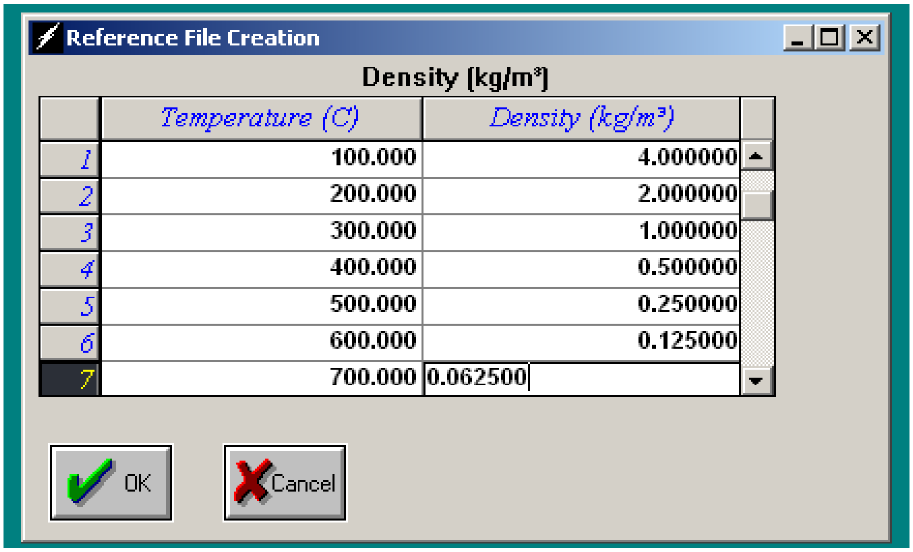


Figure 31. Density data entry table.

Step 6: Two new windows will appear. The first is a plot of the density/temperature data. The second has the tabulated data as well as curve fit data for polynomial curve fits up to fifth order. Also r, r^2, and standard deviation values are provided for each curve fit. These two windows are shown in Figures 32 and 33.

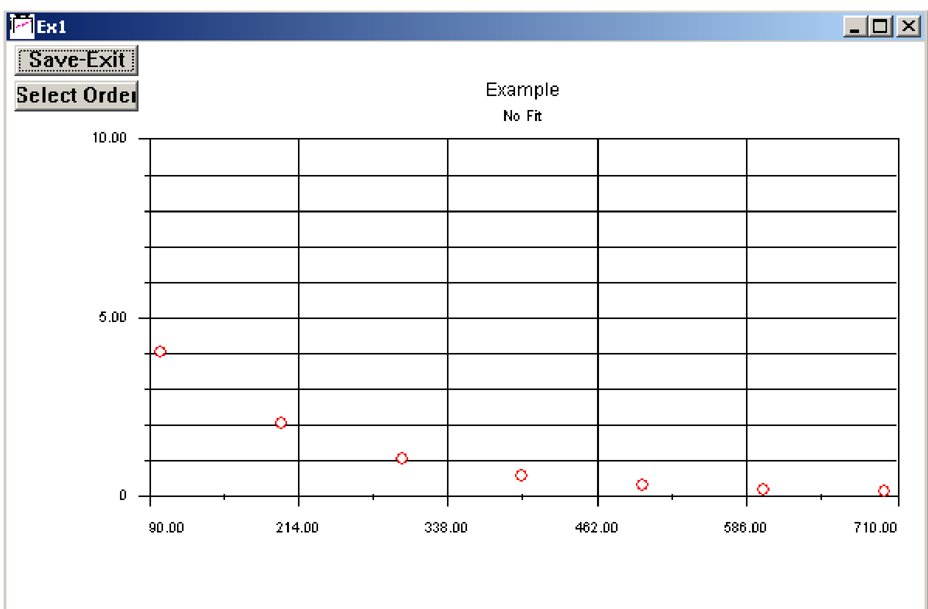
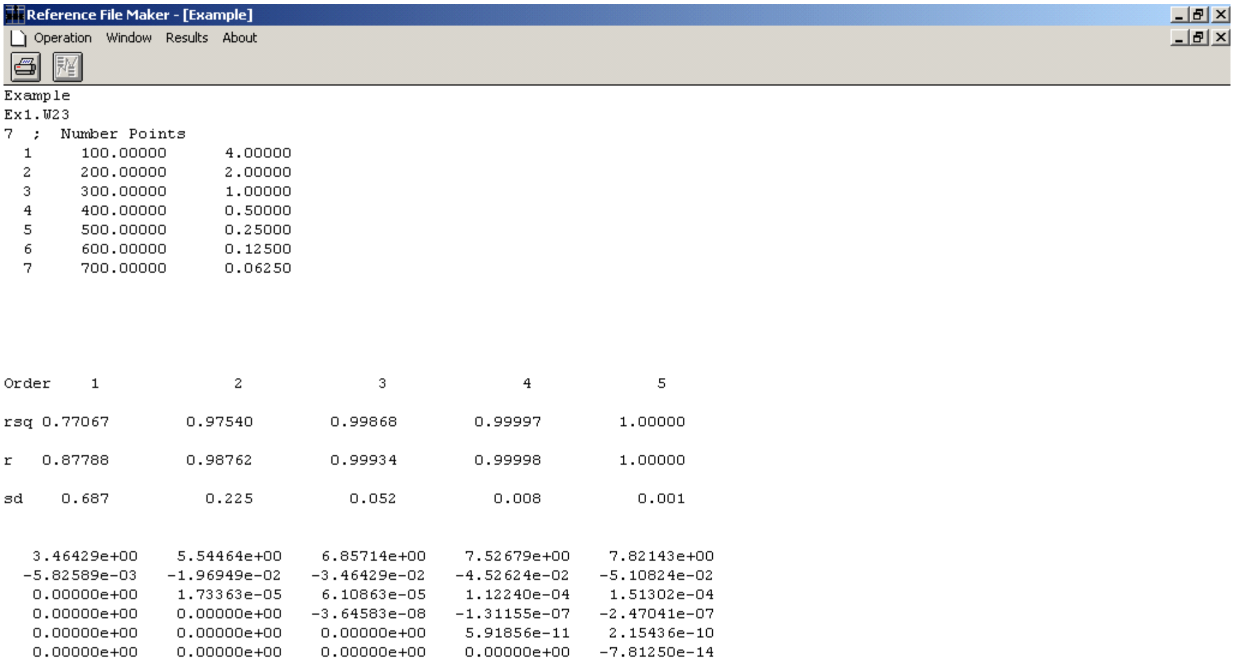


Figure 32. Density data plotted as a function of temperature.

The polynomial curve fits, as well as an interpolated one, may be plotted with the data by either using the “Select Order” function on the plot window or using the “Graphic Parameters” button (next to the printer button in Figure 33, the plot window must be open to use this button).


Reference File Maker - [Example]
Operation Window Results About

Example
Ex1.W23
7 ; Number Points

1	100.00000	4.00000
2	200.00000	2.00000
3	300.00000	1.00000
4	400.00000	0.50000
5	500.00000	0.25000
6	600.00000	0.12500
7	700.00000	0.06250

Order	1	2	3	4	5
rsq	0.77067	0.97540	0.99868	0.99997	1.00000
r	0.87788	0.98762	0.99934	0.99998	1.00000
sd	0.687	0.225	0.052	0.008	0.001
	3.46429e+00	5.54464e+00	6.85714e+00	7.52679e+00	7.82143e+00
	-5.82589e-03	-1.96949e-02	-3.46429e-02	-4.52624e-02	-5.10824e-02
	0.00000e+00	1.73363e-05	6.10863e-05	1.12240e-04	1.51302e-04
	0.00000e+00	0.00000e+00	-3.64583e-08	-1.31155e-07	-2.47041e-07
	0.00000e+00	0.00000e+00	0.00000e+00	5.91856e-11	2.15436e-10
	0.00000e+00	0.00000e+00	0.00000e+00	0.00000e+00	-7.81250e-14

Figure 33. Tabulated data and curve fit information.

Figure 34 shows the drop down menu associated with the "Select Order" button. A fifth order curve fit has been applied to the example data.

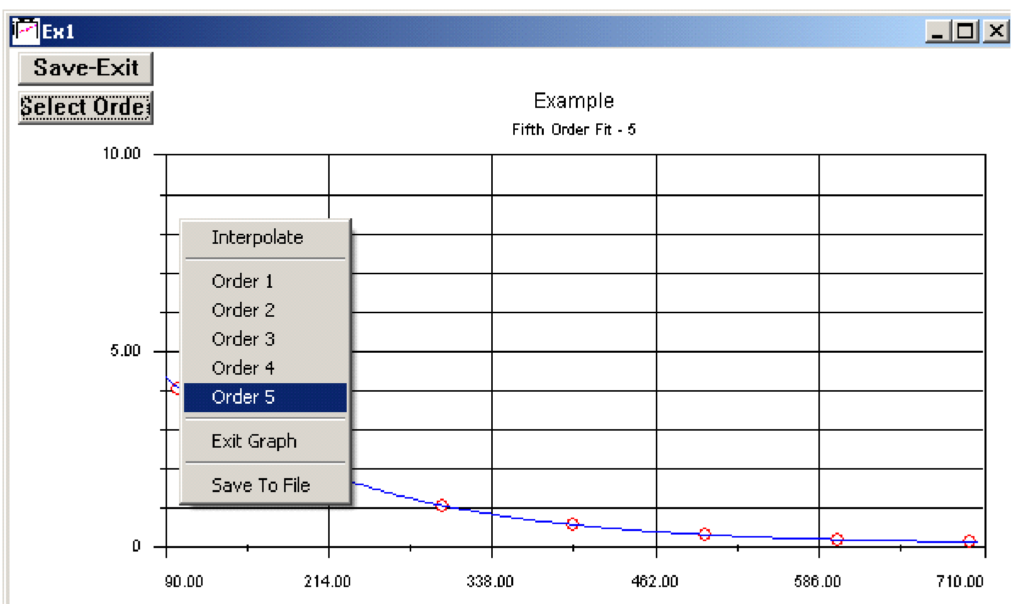


Figure 34. Plot with fifth order curve fit created using "Select Order" button.

Figure 28 shows the pop-up control associated with the “Graphic Parameters” button. To apply a curve fit the desired curve fit type is selected under “Fitting Information,” and “Curve fit through points” is selected under “Display Options.” This control window also allows labeling of the plot and scaling of the axes.

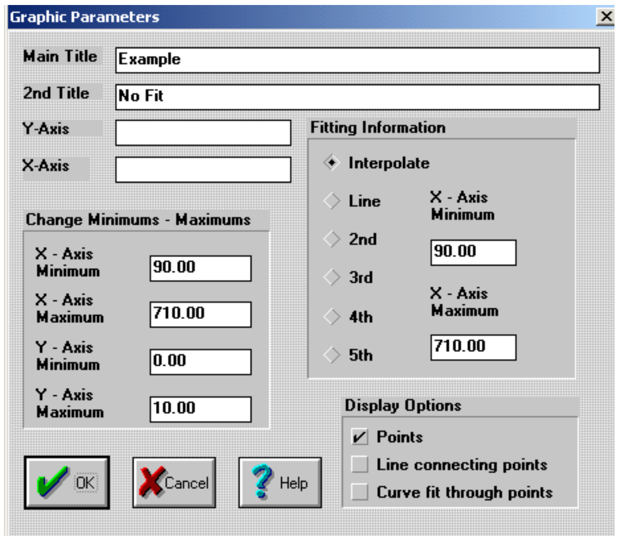


Figure 35. “Graphic Parameters” control window.

4.2. Diagnostic Functions

The Anter LaserFlash system software contains a sophisticated diagnostic capability. This diagnostic function should be used only with consultation from the vendor. If the diagnostic system is required, the vendor should be consulted. The vendor will guide the user through the process and analyze the diagnostic data.