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Technical Guidance on Use of the NETZSCH LFA 447 Nanoflash for Measurement of Ceramic–Metallic (Cermet) Pellet Specimen

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May 2022

OAK RIDGE National Laboratory

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Radioisotope Science and Technology Division

TECHNICAL GUIDANCE ON USE OF THE NETZSCH LFA 447 NANOFLASH FOR MEASUREMENT OF CERAMIC–METALLIC (CERMET) PELLET SPECIMEN

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May 2022

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ACRONYMS

IR	infrared
LN2	liquid nitrogen

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ABSTRACT

The NETZSCH LFA 447 Nanoflash instrument uses the laser flash method to measure thermal diffusivity of a material. Thermal conductivity can be determined if specific heat and density are known for that material. The Plutonium-238 Supply Program is interested in reestablishing the capability to determine thermal diffusivity and thermal conductivity of various neptunium-237 Al/cermet samples during heating. The bottom side of a plane parallel sample is heated by an energy pulse from a light source (in this case, a xenon flash lamp). An infrared detector is on the top side of the sample and detects the time-dependent temperature rise of the sample due to the energy input from the xenon lamp. The LFA 447 Nanoflash is user-friendly, simple to operate, and requires minor sample preparation, which will allow technicians to be trained easily on its use. When using the multiproperty measurement option, the LFA 447 Nanoflash can determine both thermal diffusivity and specific heat, which means only density is needed to identify the thermal conductivity of a sample. This equipment is recognized for being highly accurate and having fast test times, replacing steady-state methods that have proven to be difficult to implement and much slower.

1. NETZSCH LFA 447 NANOFLASH INSTRUMENT INSTALLATION

1.1 LFA 447 NANOFLASH INSTALLATION

1.1.1 Equipment Setup

It is essential to identify a good working space for the LFA 447 Nanoflash. For example, the area in which it is placed should ideally have a constant room temperature, no direct sunlight, and a dust-free environment. Other important details pertaining to the equipment setup are listed below.

- The work surface should be stable and mostly free from vibrations, or a shock-absorbent table should be available. Unwanted vibrations during measurement will affect the sensor and influence results.

- The LFA 447 Nanoflash will need to be near a computer as it requires a serial port to USB cable connection.

- The chiller or cooling water system should be near the equipment and placed out of the way (i.e., under the table or workspace).

- Connection of the cooling water system uses two hoses that need to be connected properly to ensure the water flow loop between the chiller and dilatometer is established (as shown in Figure 1).

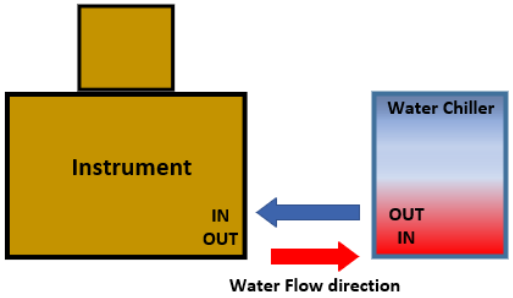


Figure 1. Setup between LFA 447 Nanoflash and chiller.

1.1.2 Instrumentation and Accessories

1.1.2.1 Extractor Tool

The extractor tool is used to lift the furnace lid up to allow access to the samples. If the furnace lid is still hot, place the lid on the scratch plate. The extractor tool may also be used to lift the sample tray out of the furnace. Figure 2 shows the extractor tool and accessories.

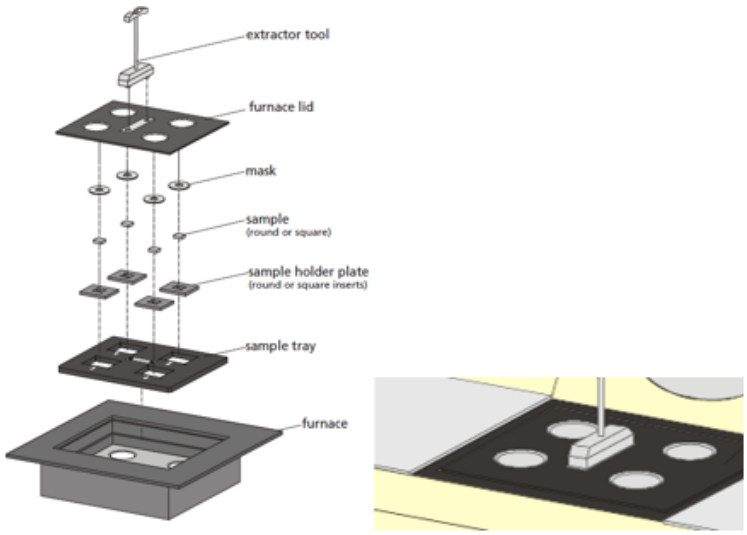


Figure 2. Extractor tool and accessories.

1.1.2.2 Sample Holder

The automatic sample changer holds up to four samples, each of which can be measured during a single test. Each sample is placed into its own sample holder plate. Four sample holder plates are placed into a sample tray and then covered by a mask (components show in Figure 2). The mask we used is made of aluminum foil and has an opening in the center to allow access to the sample. The sample holder plates used for the ^{238}Pu project allow for testing round samples that measure 12.7 mm or 6.35 mm in diameter.

1.1.2.3 Temperature Regulator

The internal heater or furnace allows the LFA 447 Nanoflash to operate in the range from room temperature up to 300°C. It is integrated into the automatic sample changer to allow for quick heating and cooling times.

A water chiller or cooling water system is needed to operate the instrument. The water chiller regulates the temperature of the instrument components involved in the measurement and cooling of the system. The water chiller's thermostat should be adjusted 2°C–3°C below room temperature.

Liquid nitrogen (LN2) is used to cool the infrared (IR) detector. Approximately 200 ml of LN2 is needed to be deposited into the detector casing.

1.1.2.4 Detector

The IR detector (as shown in Figure 3) is a liquid nitrogen–cooled In-Sb detector made from a crystalline compound of indium and antimonide. The narrow-gap semiconductor is sensitive between 1and 5 µm wavelengths. Because this is a noncontact measurement technique, response time for the signal acquisition system is short and the sample changer is automated.

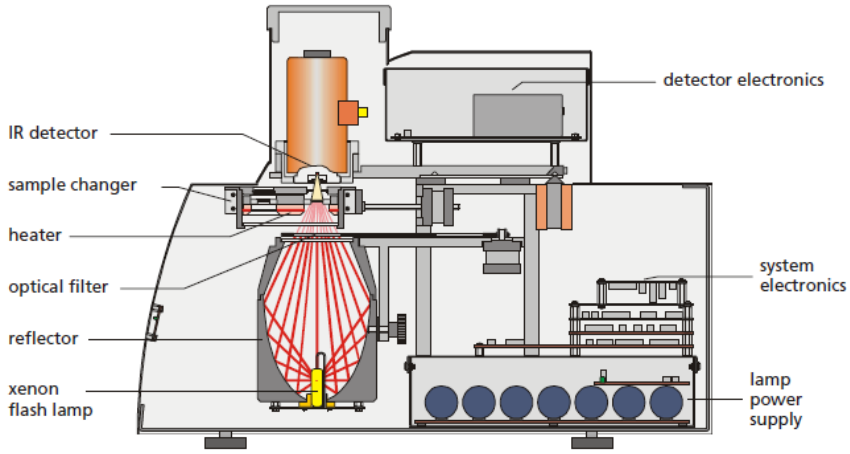


Figure 3. Internal image of the LFA 447 Nanoflash.

1.1.2.5 Flash Lamp

The xenon lamp is placed in a reflector (as shown in Figure 3), which allows the heat given off to focus on the entire sample surface. The energy released from the xenon lamp can be adjusted using the associated software, as well as the length of the pulse.

2. LFA 447 NANOFLASH SAMPLE REQUIREMENTS

2.1 SAMPLE PREPARATION

Sample preparation begins with determining the volume percent of the starting materials needed to create the samples. Samples in the form of pellets are created by weighing out the starting materials and recording the weights on the LFA thermal conductivity pellet calculations worksheet (as shown in Figure 4). In Figure 4, for example, aluminum and cerium oxide (used as a surrogate for neptunium oxide) are used to create an Al/cerium oxide cermet pellet. The powders are thoroughly mixed and then pressed using a 0.25-in.-diameter die in a single-axis hydraulic press at 5,400 lb of force. The pellet samples were heat treated at 590°C to remove the lubricant and any other organics. The LFA thermal conductivity pellet calculations worksheet may be used to assist in necessary calculations such as density and specific heat.

Calculations for Cermet Pellets

Void Density	0.001225	g/cm³	CeO_2 Molar Mass	172.115	g/mol	Binder Volumetric Sp. Heat	0 MJ/m3-K
CeO_2 Density	7.22	g/cm³	CeO_2 Heat Capacity	67.125	J/K-mol	Element Molar Mass	140.12 g/mol
Aluminum Density	2.702	g/cm³	CeO_2 Volumetric Sp. Heat	2.8158	MJ/m³-K	Aluminum Molar Mass	26.9815 g/mol
Binder Density	0.97	g/cm³	Aluminum Volumetric Sp. Heat	2.43	MJ/m³-K		
Target Vol% CeO_2	35	vol%	Void Volumetric Sp. Heat	0.001225	MJ/m³-K		

STARTING DATA

Pellet ID	Pellet Dia. (in)	Pellet Length (in)	Pellet Vol (cm³)	CeO2 Vol %	Void Vol %	Aluminum Vol %	CeO2 Mass (g)	Void Mass (g)	Aluminum Mass (g)	Wt% CeO_2 (g)	Wt% Void (g)	Wt% Aluminum (g)	Pellet Mass (g)	Pellet Density (g/cm³)	Press Force (lbs)	C_p (Sp. Heat) CeO_2 (J/g-K)	C_p (Sp. Heat) Void (J/g-K)	C_p (Sp. Heat) Aluminum (J/g-K)	C_p (Sp. Heat) Pellet (J/g-K)	Diffusivity Pellet (mm²/s)	C_v=C_p Pellet (J/g)
LFA-CEO2-35	0.25	0.08543	0.0687	35	10	55	0.1605	0.00001	0.0950	62.816%	0.003%	37.181%	0.2555	3.718	5400	0.4340	1.0120	0.9229	0.6158	11.2600	0.6158

Figure 4. LFA thermal conductivity pellet calculations worksheet.

The LFA 447 Nanoflash requires specific sample preparation with regards to sample diameter, thickness, and coating. The sample holders can accommodate round samples that have a diameter of either 12.7 mm or 6.35 mm. The samples must be prepared in such a way as to obtain flat and parallel surfaces to accurately determine the thickness of the sample. The sample should be 1 mm–3 mm thick. Samples with lower diffusivity should be 1 mm–1.5 mm thick. Samples with a medium diffusivity should be 1.5 mm–2 mm thick. Samples with a high diffusivity should be 2 mm–3 mm thick.

Samples always need to be coated with a layer of graphite because the analysis of the flash diffusivity data requires that the energy be absorbed at the surface of the sample. The graphite layer will enhance the absorption of the xenon's energy; likewise, the IR detector measures the sample's surface temperature, and the graphite layer will enhance the emission of IR radiation to the detector.

2.1.1 Creating the Sample Pellet

1. Weigh out the desired amount of aluminum powder.
2. Weight out the desired amount of cerium oxide powder.
3. Thoroughly mix the powders together.
4. Lubricate the die components and plunger with 5% stearic in chloroform and allow them to dry for a few minutes.
5. Carefully pour the mixture into a 0.25-in.-diameter die using a funnel to aid in this step.
6. Using a single-axis hydraulic press, subject the sample to 5,400 lb of force for 3 min.
7. Measure the sample's diameter with a micrometer.
8. Measure the sample's thickness with a micrometer.
9. Repeat the measurement of the sample's thickness at two other places on the sample.
10. Calculate the average of the three measurements and use that as the value for the thickness of the sample.
11. Calculate the density of the pellet.
12. Calculate the specific heat of the pellet.

2.1.2 Coating the Sample in Graphite

1. Perform the graphite sample coating work in a fume hood or other well-ventilated area.
2. Clean the sample with a solvent.
3. Place the samples into a cardboard container.
4. Shake the graphite can and hold it ~8–10 in. from the samples to be coated.
5. Spray the samples with a slow sweeping motion across the samples several times to obtain a thin coating.
6. Wait 2–5 min. for the graphite coating to dry.
7. Repeat Steps 4–6 as many times as needed to thoroughly coat with graphite.
8. Carefully turn the samples over to coat the other side, making sure not to damage the first side.
9. Repeat Steps 4–6 as needed to coat the remaining side.

3. LFA 447 NANOFLASH INSTRUMENT PREPARATION

3.1 PREPARING THE IR DETECTOR

3.1.1 Preparing the IR Detector

1. Don appropriate personal protective equipment (e.g., face shield and cryogenic gloves) prior to handling the LN2.
2. Obtain 500 mL of LN2.
3. Remove the IR detector's gray cover.
4. Remove the plug from the top of the IR detector's casing.
5. Insert the funnel into the opening.
6. Carefully fill the funnel with LN2.
7. Continue to add LN2 slowly, allowing time for the LN2 to cool the components.
8. Once there is ~200 mL of LN2 in the reservoir, wait 5 min. for the detector to stabilize.
9. Remove the funnel.
10. Replace the plug and the IR detector's gray cover.
11. Periodically refill the LN2 as needed. If LN2 has become too low, the software will alert the user to fill the LN2.

4. RUNNING THE LFA 447 NANOFLASH INSTRUMENT

4.1 DATA COLLECTION AND EVALUATION

4.1.1 Navigating the Software to Set Up a Sample Run

The LFA 447 Nanoflash has a comprehensive package of software that offers a suite of tools supporting the instrument's measurement functions through a program called Nanoflash and the instrument's analysis functions through a program called LFA Analysis. The software can simultaneously complete measurements and data analysis. For the Nanoflash program to run properly, a chiller must be connected to the instrument and turned on. The chiller functions as a thermostat controlling the LFA's relevant components to the measurement and cooling of the furnace system. Follow the steps below to set up a sample run.

1. Turn on the chiller and set it a few degrees below room temperature (e.g., 20°C).
2. Ensure the IR detector has been prepared with LN2.
3. Open the Nanoflash program using the desktop icon shown in Figure 5.

Figure 5. Nanoflash desktop icon.

4. Click Okay after the Connect to Nanoflash window has appeared and has detected the instrument (as shown in Figure 6).

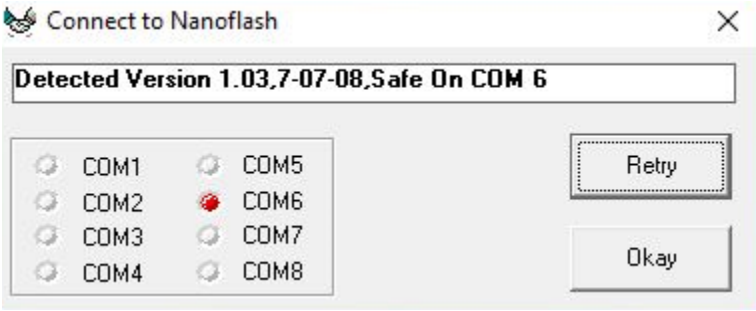


Figure 6. Connect to Nanoflash window with instrument detected.

- If communication with the hardware has failed, the message shown in Figure 7 will appear in the Connect to Nanoflash window.

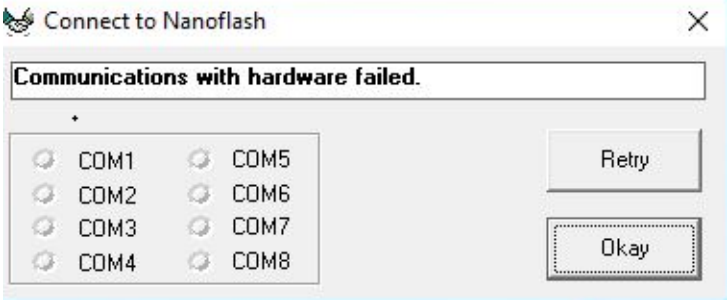


Figure 7. Communications failed message.

- To resolve the communication failure, check the following:
  - Is the instrument turned on?
  - Is the instrument properly connected to the computer via USB?
  - Is the chiller connected?
  - Is the chiller working properly?

- After resolving the problem, click Retry. A window will appear that shows it is searching for the instrument connection (as shown in Figure 8).

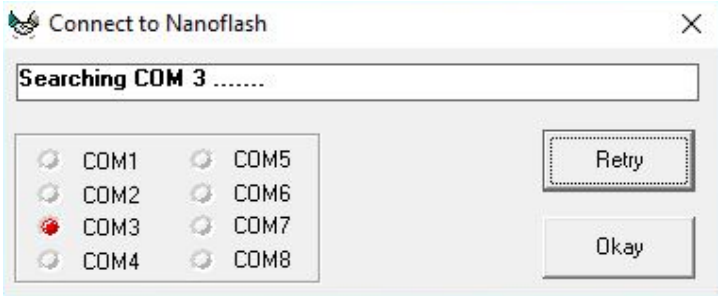


Figure 8. Searching for connection message.

- A pop-up window will appear momentarily stating the instrument is rebooting (as shown in Figure 9).

Figure 9. Rebooting pop-up window.

- Nanoflash will open to the main screen (as shown in Figure 10).

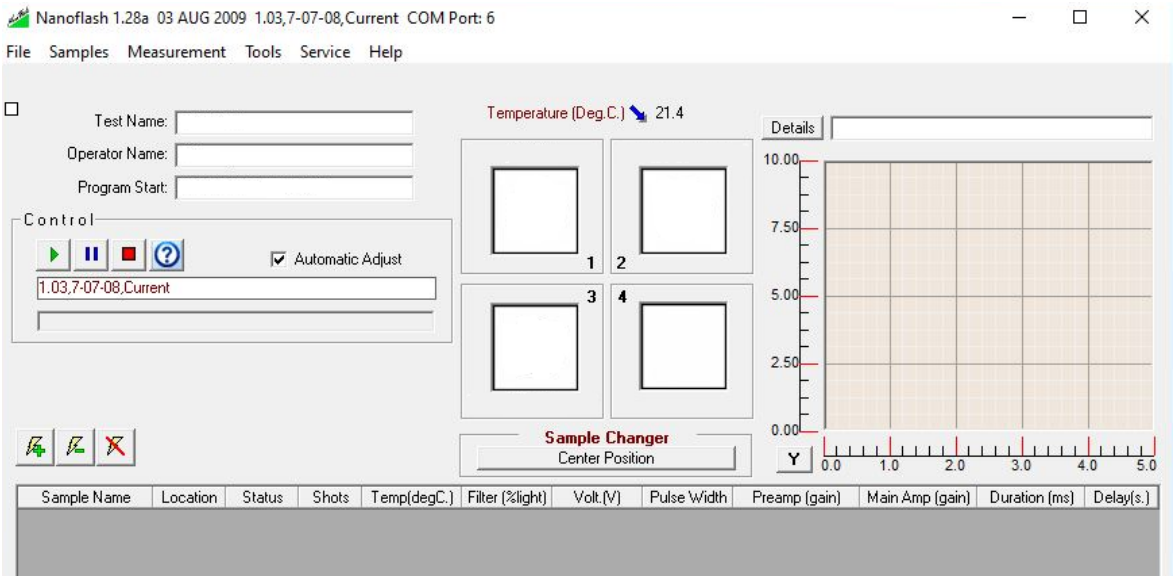


Figure 10. Nanoflash main screen.

5. On the main screen, enter the test name and operator name. The program start will auto-populate, as shown in Figure 11.

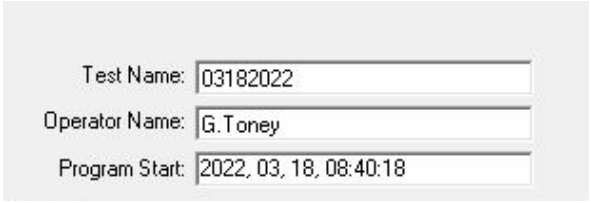


Figure 11. Basic test information.

6. Select Instrument Configuration from the File tab (as shown in Figure 12, left panel).

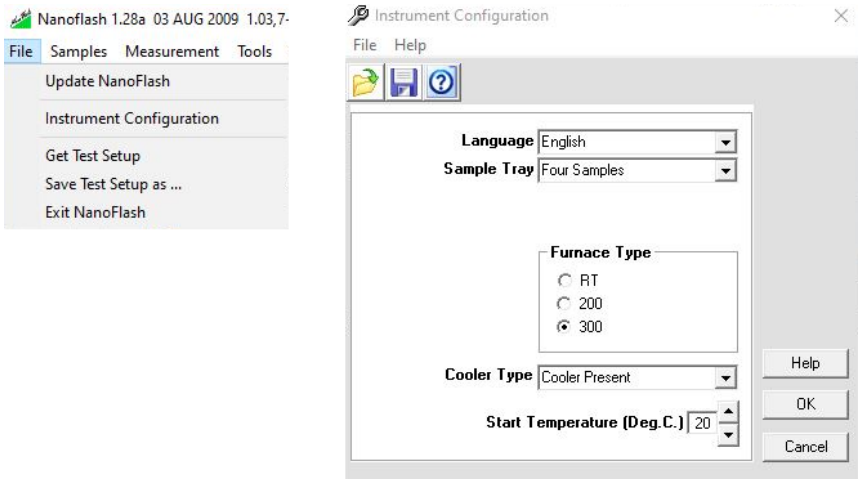


Figure 12. Instrument configuration settings.

- Ensure the instrument is configured properly with the sample tray set to four samples, the furnace type set to 300, cooler type shows cooler present, and the start temperature is set a few degrees below room temperature (usually 20°C) (as shown in Figure 12, right panel).

7. On the main screen, find the sample information under the temperature settings (as shown in Figure 13). Note in the figure the four samples have not been defined so there is no information being populated.

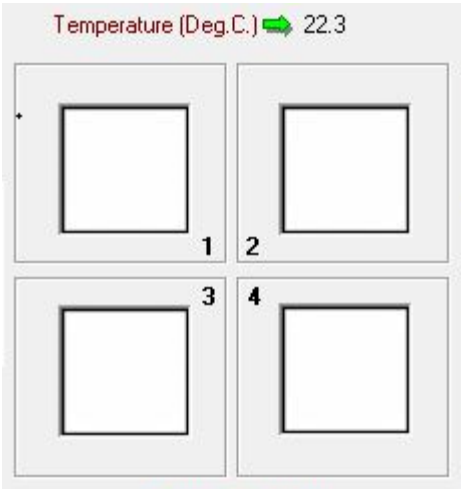


Figure 13. Sample configuration and informational display.

- To enter the sample information, right-click on one of the empty sample boxes or select Sample Definition from the Samples tab (as shown in Figure 14).
- Choose 1 through 4 to start entering the information for each sample (as shown in Figure 14).

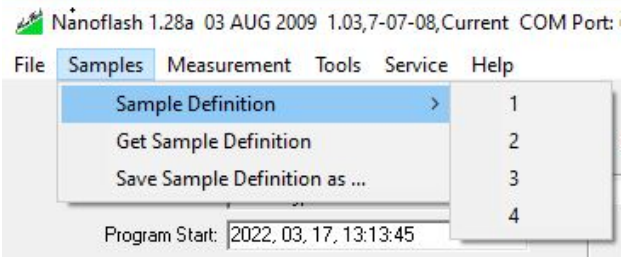


Figure 14. Sample definition using the Sample tab.

- Select the sample holder that matches the one being used (e.g., 10.0rd is used for the smallest round sample holder).
- Enter the sample name and any remarks on the sample definition form (as shown in Figure 15).

- Ensure Single Layer is selected for the type of measurement.
- Enter the sample diameter in millimeters.
- Enter the material type, thickness in millimeters, density in g/cm^3, temperature, and cp in J/g⋅K.
- Select Define when all the information has been entered into the sample definition form.

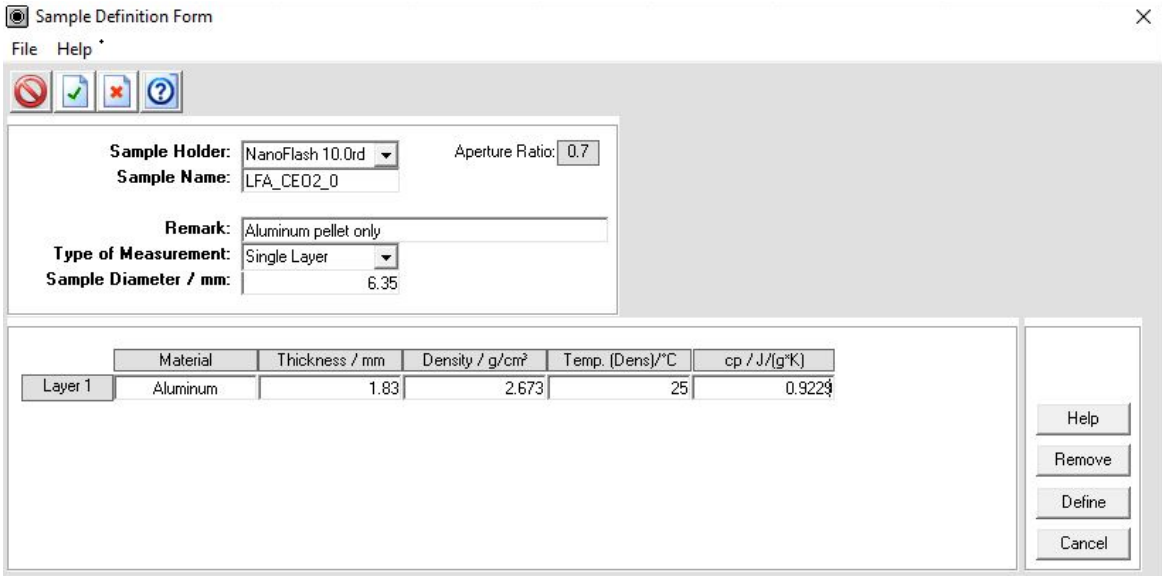


Figure 15. Sample definition form.

8. On the main screen, the sample information should be populated after all the sample information has been entered on the sample definition forms (as shown in Figure 16).

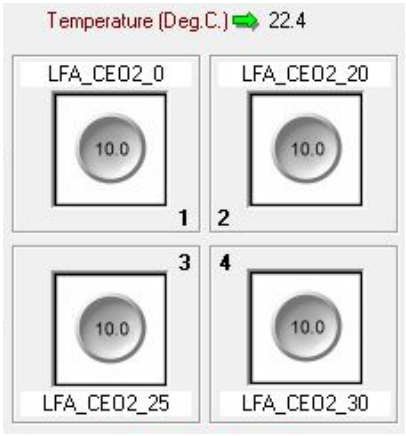


Figure 16. Sample information populated on the main screen.

9. On the main screen, select Measurement Parameters from the Measurement tab (as shown in Figure 17).

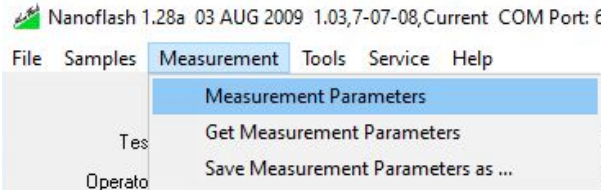


Figure 17. Measurement parameters selected using the Measurement tab.

- Under "Automatic Adjust," ensure the number of Optimization Retries is set to 10, recovery attempts are set to 3, and Autobreak After is set to120 min. (as shown in Figure 18).
- Leave the Stability Criteria at a baseline of 300 points, equilibrium at 1 min., and tolerance at 0.k ± K.
- Under Standard Samples, select the material from the drop-down box.
- Enter the thickness of each sample in millimeters and select Accept.
- Select OK to return to the main screen.

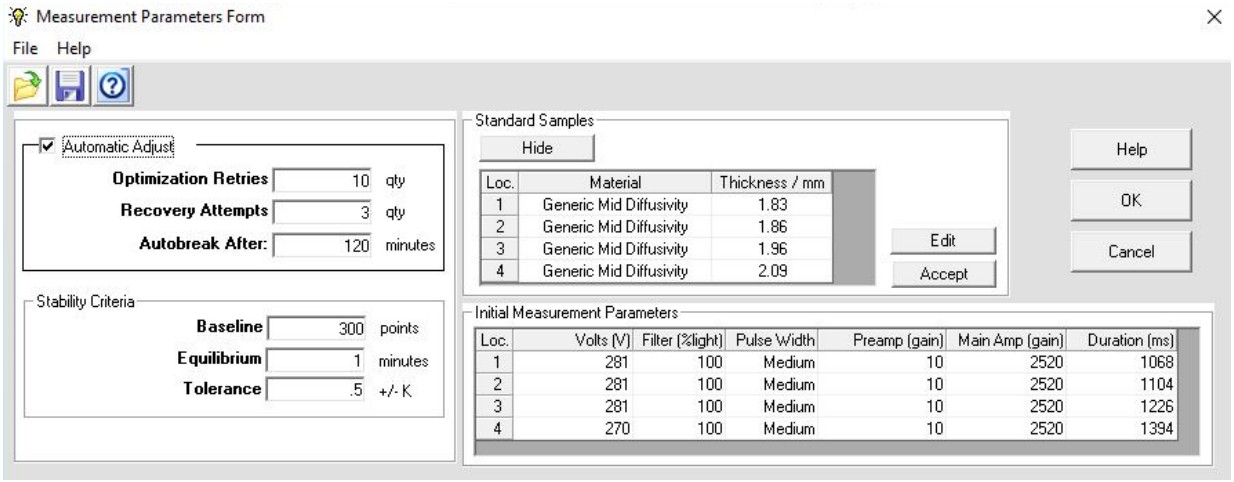


Figure 18. Measurement parameters form with information populated.

10. Select the Add Shots button to open and set up the temperature program.

- The samples that need to be run must be selected individually from the drop-down box, or all samples can be selected.
- Create a temperature program (as shown in Figure 19) by entering the starting temperature (25°C), the number of steps to measure at different temperatures (5), the increase in temperature between the steps (25°C), the number of shots (at least 3), and the delay (1–3 min.).

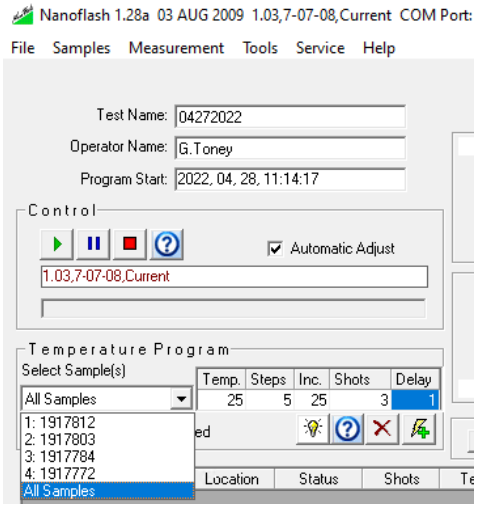
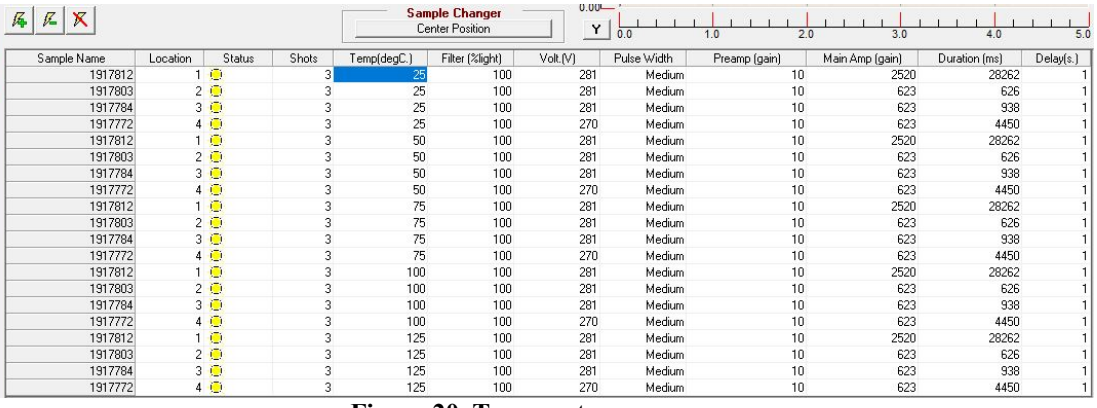


Figure 19. Temperature program information.

- Select the Add Shots button below the delay settings to add the created temperature program to the sample queue (as shown in Figure 20).
- In Figure 20, all the samples were selected from the drop-down box; 25°C was selected as the starting temperature, 5 steps were selected (therefore, the range is 25 to 125°C), 25°C was selected as the increase in temperature for each step, 3 shots were selected (i.e., the instrument will acquire 3 acceptable shots), and a 1 min. delay was selected (i.e., the instrument will wait 1 min. after each shot).



Sample Name	Location	Status	Shots	Temp(degC.)	Filter (%light)	Volt.(V)	Pulse Width	Preamp (gain)	Main Amp (gain)	Duration (ms)	Delay(s.)
1917812	1		3	25	100	281	Medium	10	2520	28262	1
1917803	2		3	25	100	281	Medium	10	623	626	1
1917784	3		3	25	100	281	Medium	10	623	938	1
1917772	4		3	25	100	270	Medium	10	623	4450	1
1917812	1		3	50	100	281	Medium	10	2520	28262	1
1917803	2		3	50	100	281	Medium	10	623	626	1
1917784	3		3	50	100	281	Medium	10	623	938	1
1917772	4		3	50	100	270	Medium	10	623	4450	1
1917812	1		3	75	100	281	Medium	10	2520	28262	1
1917803	2		3	75	100	281	Medium	10	623	626	1
1917784	3		3	75	100	281	Medium	10	623	938	1
1917772	4		3	75	100	270	Medium	10	623	4450	1
1917812	1		3	100	100	281	Medium	10	2520	28262	1
1917803	2		3	100	100	281	Medium	10	623	626	1
1917784	3		3	100	100	281	Medium	10	623	938	1
1917772	4		3	100	100	270	Medium	10	623	4450	1
1917812	1		3	125	100	281	Medium	10	2520	28262	1
1917803	2		3	125	100	281	Medium	10	623	626	1
1917784	3		3	125	100	281	Medium	10	623	938	1
1917772	4		3	125	100	270	Medium	10	623	4450	1

Figure 20. Temperature program.

- In Figure 20, the status listed for each sample is yellow, meaning the sample has not been completed. After the sample has run, the status will change to green (as shown in Figure 21).

Sample Name	Location	Status	Shots	Temp(degC.)
LFA_CEO2_0	1	5 - 1	5	150
LFA_CEO2_20	2	5 - 1	5	150
LFA_CEO2_25	3	5 - 1	5	150
LFA_CEO2_0	1	5 - 1	5	200

Figure 21. Sample status.

11. Begin running the samples by selecting the Start icon under the Control section of the main screen (as shown in Figure 22).

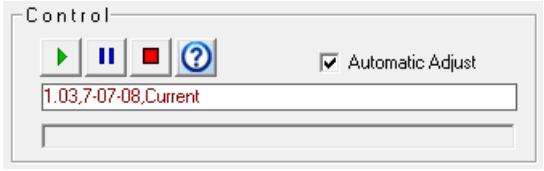


Figure 22. Control buttons for the sample run.

- If it is necessary to pause the sample runs, press the Pause icon .
- If it is necessary to stop the sample runs, press the Halt icon .

4.1.2 Navigating the Software to Evaluate the Sample Data

1. Open the LFA Analysis program to evaluate the sample data using the NETZSCH desktop icon (as shown in Figure 23).

Figure 23. Analysis program icon.

2. Open the LFA Analysis shortcut found in the NETZSCH programs folder window (as shown in Figure 24).

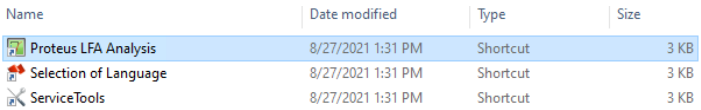


Figure 24. Open Proteus LFA analysis program.

- The Proteus LFA Analysis Logo window will flash prior to the main screen (as shown in Figure 25).

Figure 25. LFA analysis logo window.

- The main screen will open to an empty analysis (as shown in Figure 26).

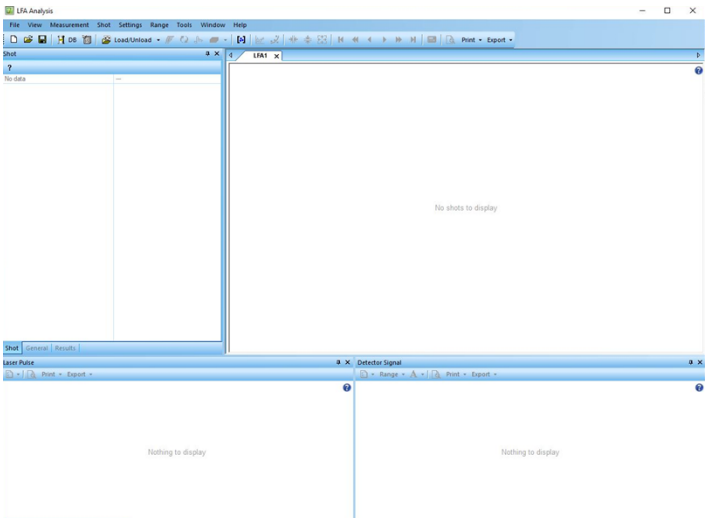


Figure 26. Empty analysis main screen.

3. Create a new database.

   - Select the Explore Database icon to create a new database.
   - Select New Database from the drop-down menu (as shown in Figure 27).

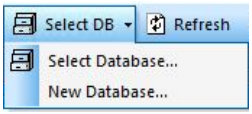


Figure 27. Select an existing or create a new database.

   - Enter a name for the database in the file name field and select Save (as shown in Figure 28).

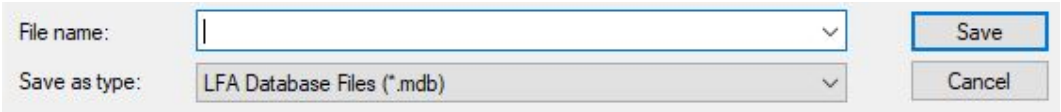


Figure 28. Create a name for the new database.

   - The database that was saved is now opened on the main screen (as shown in Figure 29).

Figure 29. Newly saved database, CeO2 samples.

4. Import LFA 447 files into the database.

- Click on the Import Files icon and select Import LFA 447 File from the drop-down menu (as shown in Figure 30).

Figure 30. Import files into the database from drop-down menu.

- A pop-up window will appear twice stating the directory does not exist. Click OK each time (as shown in Figure 31).

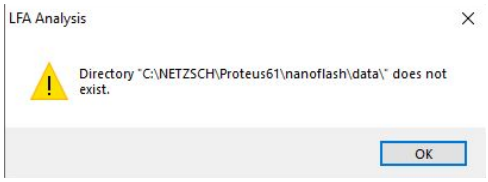


Figure 31. LFA Analysis pop-up window.

- Select the files from the correct directory, which is C:\ngbwin\ta\nanoflash\files (as shown in Figure 32).

Figure 32. The directory to use to select the saved data files.

- Select the file(s) to be imported and click Open (as shown in Figure 33).

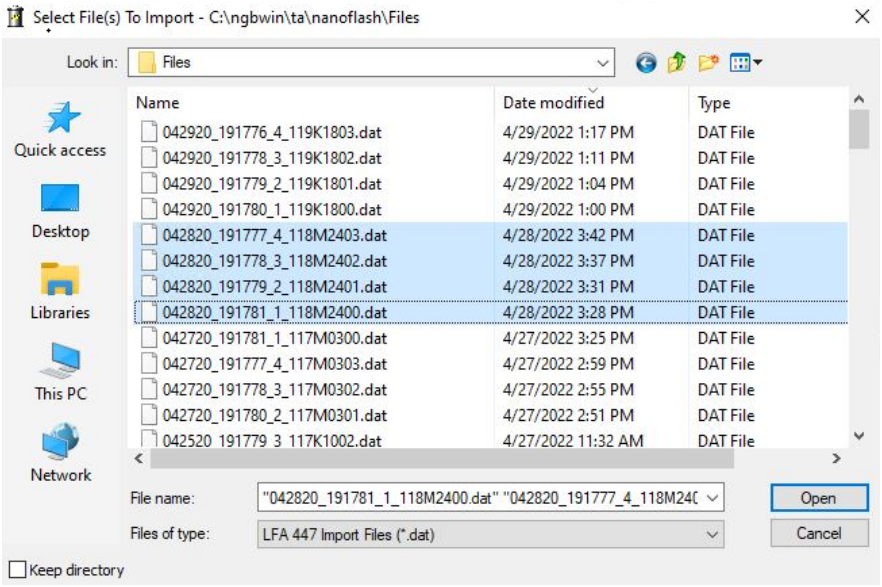


Figure 33. Highlighted files to be imported into the selected database.

- The Import Material Selection window for the sample will pop up; the material will need to be defined for the first time or selected from an existing list (as shown in Figure 34).

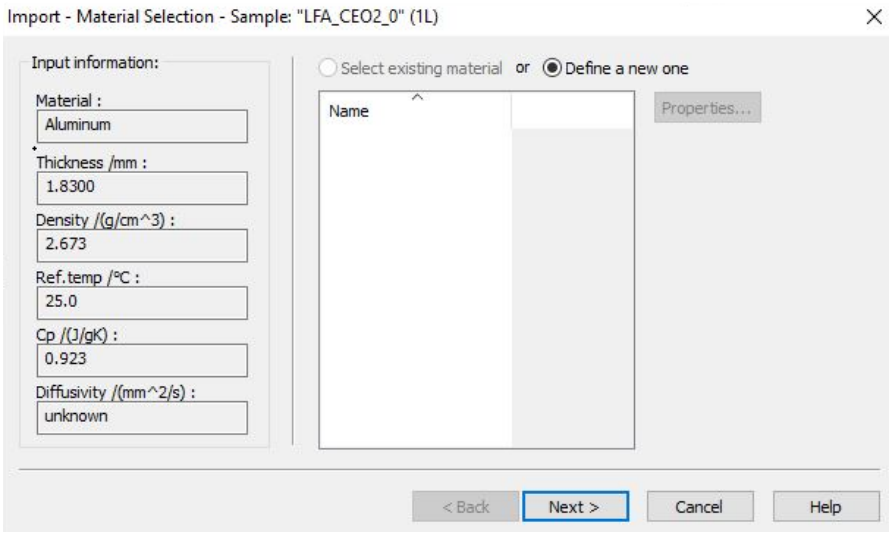


Figure 34. Import Material Selection pop-up window.

- Complete the material definition information as needed (as shown in Figure 35).
- Select Finish.

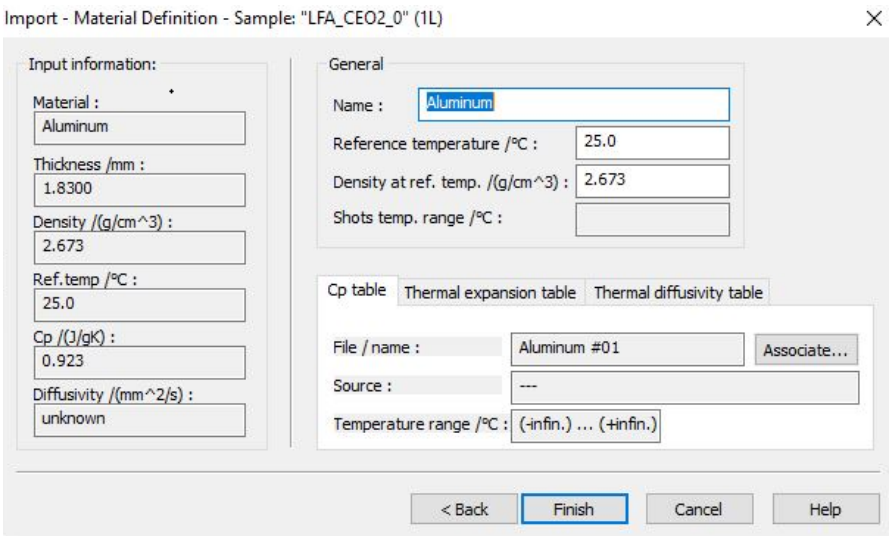


Figure 35. Complete the material definition information.

- The Import Statistics window will pop up stating the number of new measurements and the new shots to import from the files selected (as shown in Figure 36).

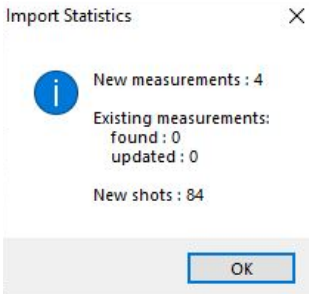


Figure 36. Import Statistics pop-up window.

5. Load the LFA files into the database.

- Highlight the files to load and select the Load icon (as shown in Figure 37).

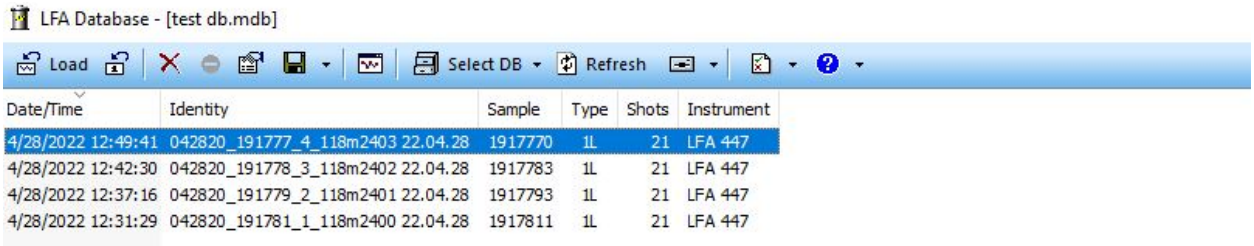


Figure 37. Files to load into the database.

6. Calculate the selected shots in the database.

- Select Yes to get correct results when the pop-up window appears (as shown in Figure 38).

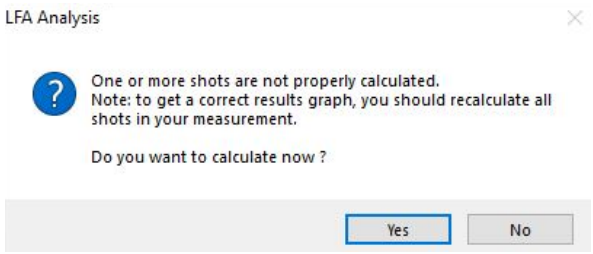


Figure 38. Calculate the selected shots pop-up window.

- Check the Cowan + pulse correction model and select OK (as shown in Figure 39).

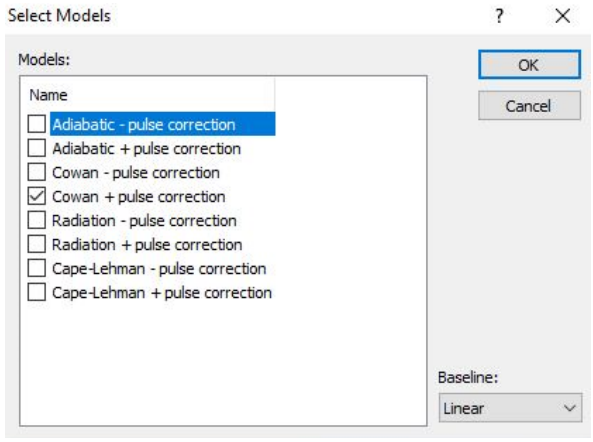


Figure 39. Select Cowan + pulse correction model.

- A pop-up window will display the shots being calculated (as shown in Figure 40).

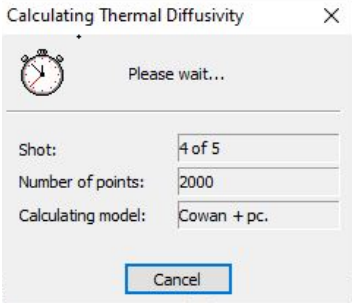


Figure 40. Shots being calculated pop-up window.

7. Calculate thermal conductivity.

- Open the Measurement tab and select Calculate Thermal Conductivity from the drop-down menu (as shown in Figure 41).

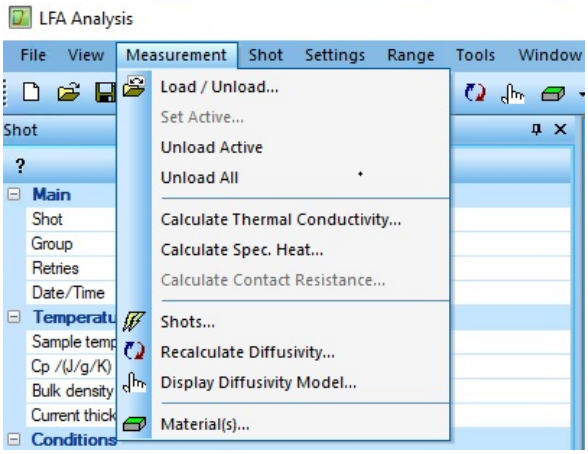


Figure 41. Calculate thermal conductivity from the Measurement tab.

- Uncheck the thermal diffusivity box, check the thermal conductivity box, and select OK (as shown in Figure 42).

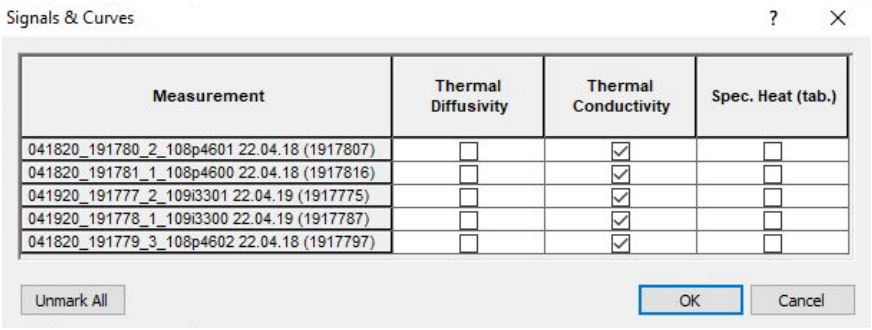


Figure 42. Calculate thermal conductivity for selected measurements.

- A pop-up window will appear asking if you want to calculate based on one Cp value; select Yes (as shown in Figure 43).

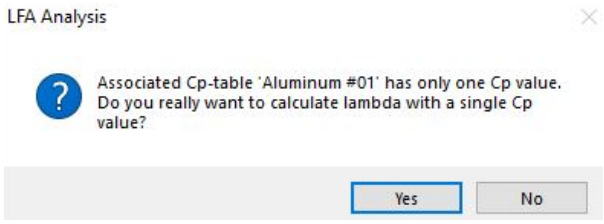


Figure 43. Verify calculation based on Cp value.

- The thermal conductivity of each sample loaded will be populated (as shown in Figure 44).

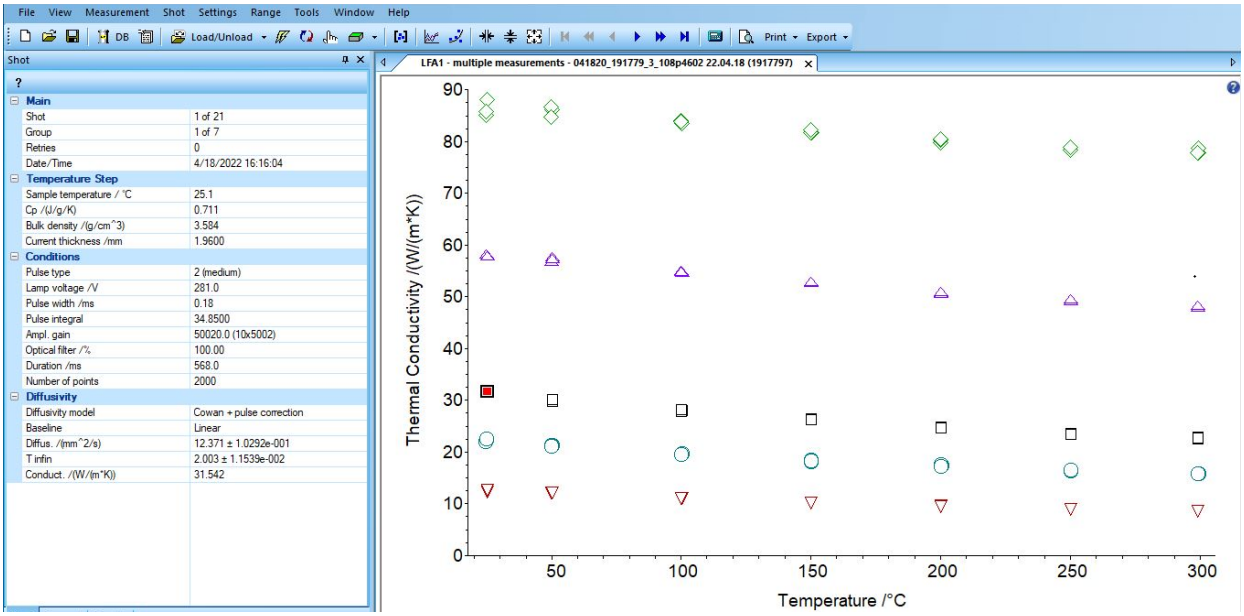


Figure 44. Thermal conductivity graph of each sample loaded in the database.

4.1.3 Exporting the Data

Data can be exported in many different formats that can be used in Excel (e.g., .jpg, .pdf, .txt).

1. To export data as a PDF, open the Export tab and select “To PDF” (as shown in Figure 45).

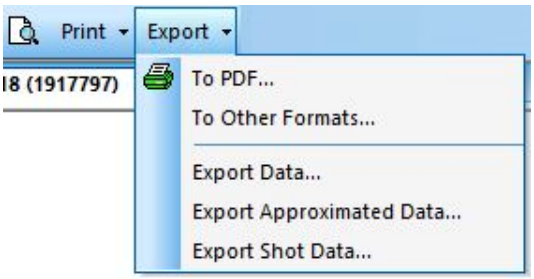


Figure 45. Export data to PDF using the Export tab.

2. Save the file as a pdf (as in Figure 46).

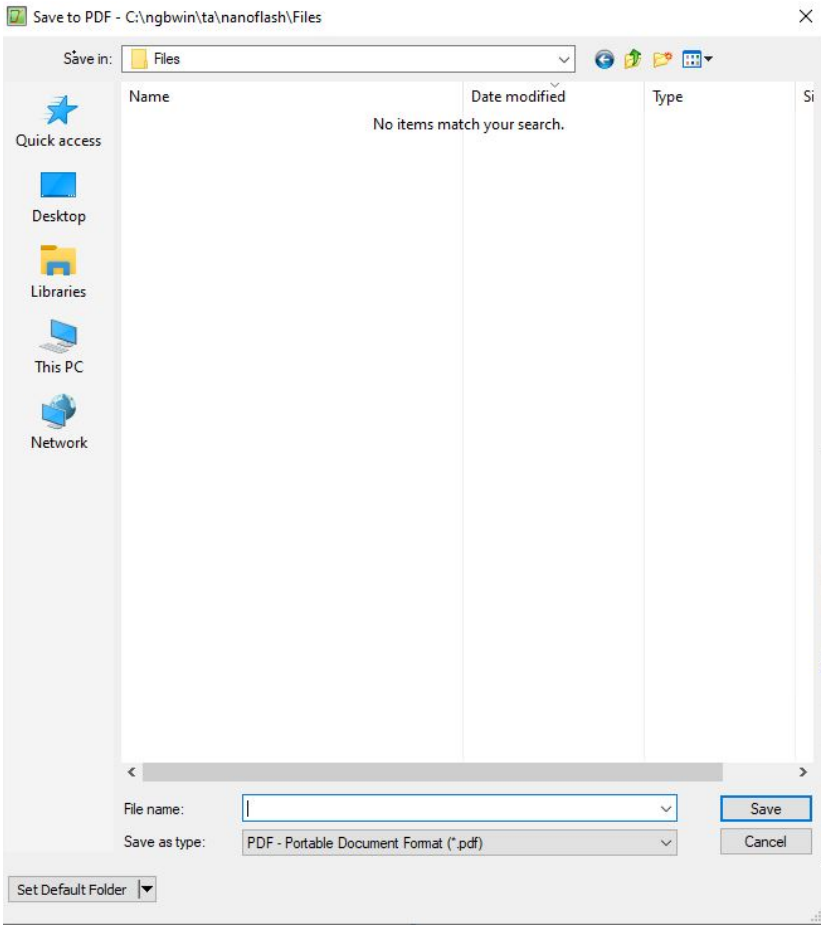


Figure 46. Save the file as a .pdf file.

3. To export the data as a .txt file to use in Excel, open the export tab, and select "Export Data" (as shown in Figure 45).

    - Verify single results and relative are selected (as shown in Figure 47).

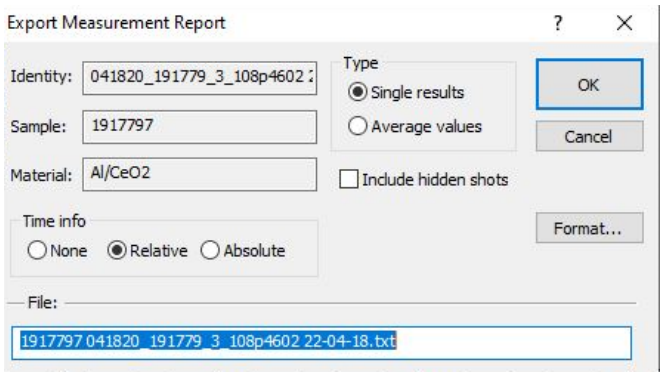


Figure 47. Export data as a .txt file.

- Click on Format and make sure the following are selected (as shown in Figure 48):

    - Custom for separators.

- Tab for Column separator.
- Point for decimal symbol.

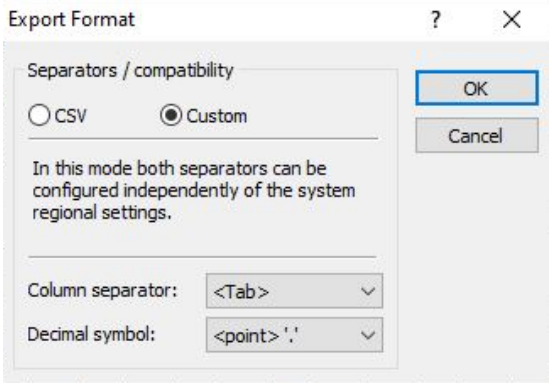


Figure 48. Export format details.

- Select OK on both pop-up windows.

4. Save the file as a .txt file.
5. Open the .txt file and copy the data into an Excel file.
    - After opening the .txt file, use Ctrl + A to select all the data.
    - Use Ctrl + C to copy the information.
    - Open Excel and use Ctrl + V to paste the information into Excel.
    - Create a data table in Excel using the mean temperature and thermal conductivity data for the samples (as shown in Figure 49).

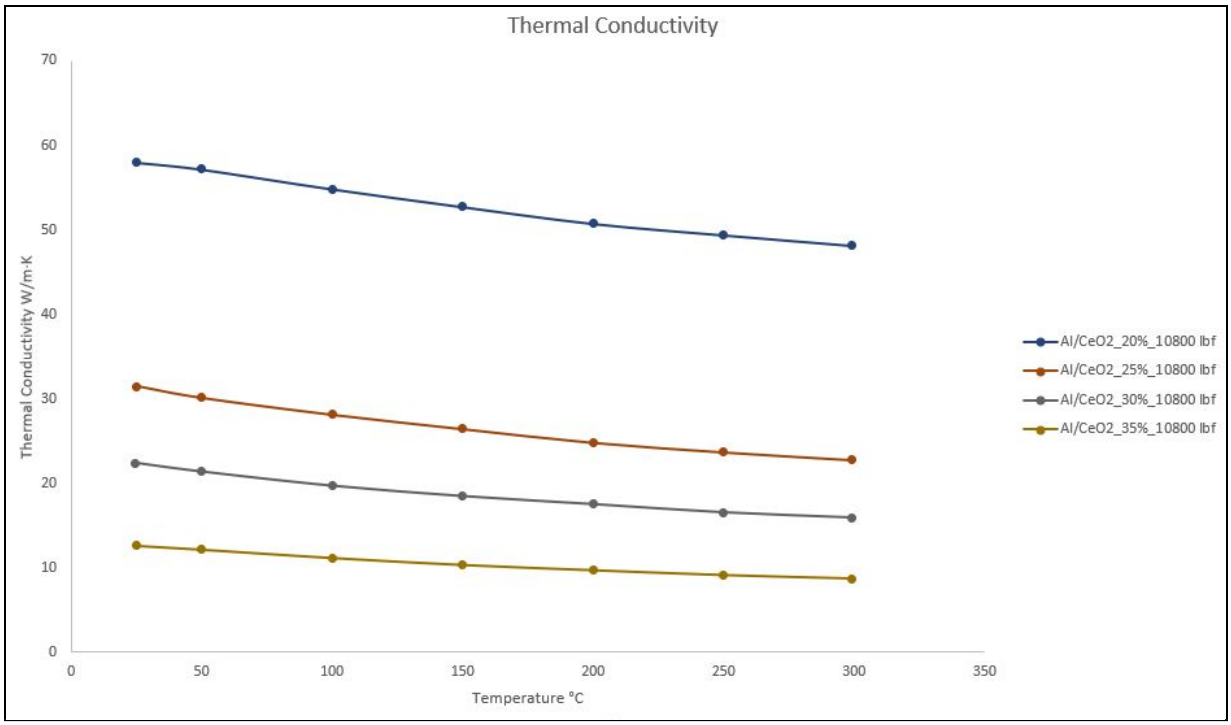


Figure 49. Thermal conductivity graph created in Excel.