Standard Operating Procedure

Use of Netzsch LFA-457 System

Facility: LFA457 System
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Scope: This SOP details the general procedure for operation of the departments Netzsch LFA-457 system.

Last Revision: 12/6/2010

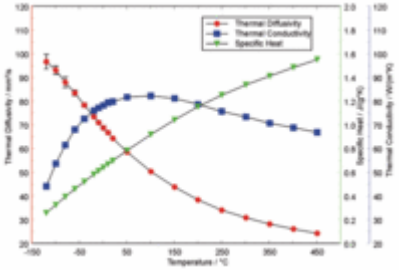
Introduction:

The NETZSCH LFA 457 MicroFlash® complies with the latest technology for modern laser flash systems. The table-top instrument allows measurements from -125°C to 1100°C using two different user-exchangeable furnaces. The innovative infrared sensor technology employed in the system enables measurement of the temperature increase on the back surface of the sample, even at temperatures of -125°C. The instrument can be used for small and large sample sizes of up to 25.4 mm diameter and, with the integrated sample changer, measurements can be run on several samples at the same time.
The vacuum-tight design enables tests under defined atmospheres.
The vertical arrangement of the sample holder, furnace and detector simplifies sample placement and, at the same time, guarantees an optimum signal-to-noise ratio of the detector signal.

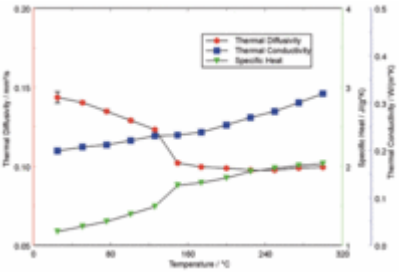
Applications:

Polycrystalline Graphite Graphite materials are known to show a maximum thermal conductivity around room temperature, which can easily be analyzed using the low temperature version of the LFA 457 MicroFlash®. The physical explanation for this maximum is the high Debye temperature of this material (> 1000 K). The decrease in thermal diffusivity with increasing temperature dominates the temperature dependence of the thermal conductivity in the high temperature region. The specific heat decreases strongly at

temperatures below room temperature and dominates the temperature dependence of the thermal conductivity there.



Polycarbonate Polycarbonate (PC) is a popular polymer material used among other things, for electric tool casings. To optimize the production/molding process by finite element simulations, the thermophysical properties have to be known. The thermal diffusivity can be determined not only in the solid region but also at temperatures above the glass transition (> 140°C) if a molten material cell is employed in the LFA 457 MicroFlash®. Together with the specific heat (measured with a DSC) and density data, the thermal conductivity can be determined. The slight increase in the thermal conductivity versus temperature is typical for 100% amorphous materials. Furthermore, the glass transition is visible in the specific heat curve and in the thermal diffusivity result. In the thermal conductivity result, this second order transition cannot be seen.

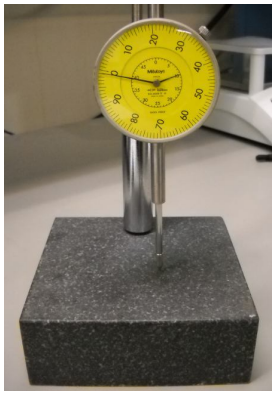


System and Experiments:

- Heating- and cooling rates: 0,01 K/min ... 50 K/min
- Laser power 15 J/pulse, (adjustable power)
- Contactless measurement of temperature rise with IR detector
- Measuring range: 0.01 mm2/s ... 1000 mm2/s (thermal diffusivity)
- Measuring range: 0.1 W/mK ... 2000 W/mK (thermal conductivity)
- Sample dimensions: 10 mm ... 25.4 mm diameter (also 8x8 mm and 10x10 mm, square) 0.1 mm ... 6 mm thickness
- Sample holder: SiC, graphite
- Liquid metal holder : sapphire
- Sample holder for liquids: platinum
- Atmosphere: inert, oxidizing, reducing, static, dynamic
- Vacuum-tight assembly up to 10-2 mbar (1 Pa)

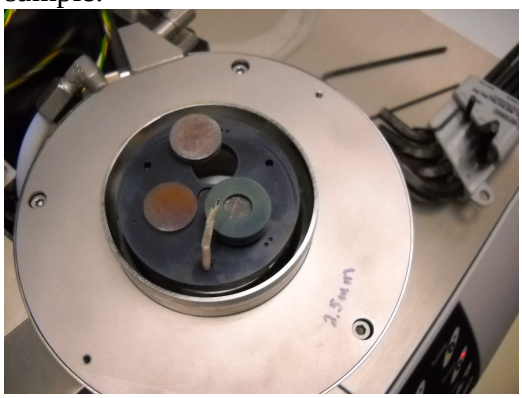
Typical Operation:

1) Turn on power to the system (no particular turn on sequence) and allow system to warm up for approximately 2 hours.
2) Fill the detector with liquid nitrogen. After the liquid nitrogen spills out around the sides of the funnel, wait for a minute or two and watch – there should be an increase in gas discharge from the chamber just before the liquid nitrogen settles down. After you see this discharge, then put on the chamber plug (if you put it on before, then it could be blown off by the discharge).
3) Samples should have been prepared with parallel surfaces, 1-4mm thick sample, and we have several sample holders which can accommodate the following sample x-y dimensions:
   12.7mm diameter qty-3
   10mm × 10mm qty-2
   8mm × 8mm qty-2
   25.4mm diameter qty-1
4) Measure the thickness of each sample in 5 locations on each sample using the high accuracy micrometer. Record these measurements, find the average thickness, and standard deviation.



5) Clean each sample using isopropyl alcohol or other appropriate cleaning agent, and a wipe.
6) Paint each sample with graphite (2-3 spray passes over the samples, allow to dry before applying subsequent coats, read instructions on the spray can). Then flip the samples (careful, the graphite wipes off easily), and paint the other side.

7) Open the furnace by depressing the button on the right side of the instrument and the down button on the front of the LFA-457 simultaneously. System will stop when it reaches full open. The furnace and detector assembly can then be rotated to the left (clockwise looking down) to get it out of the way.
8) Place samples in positions 1, 2, or 3 (assuming this sample stage is used) and record the position of each sample.

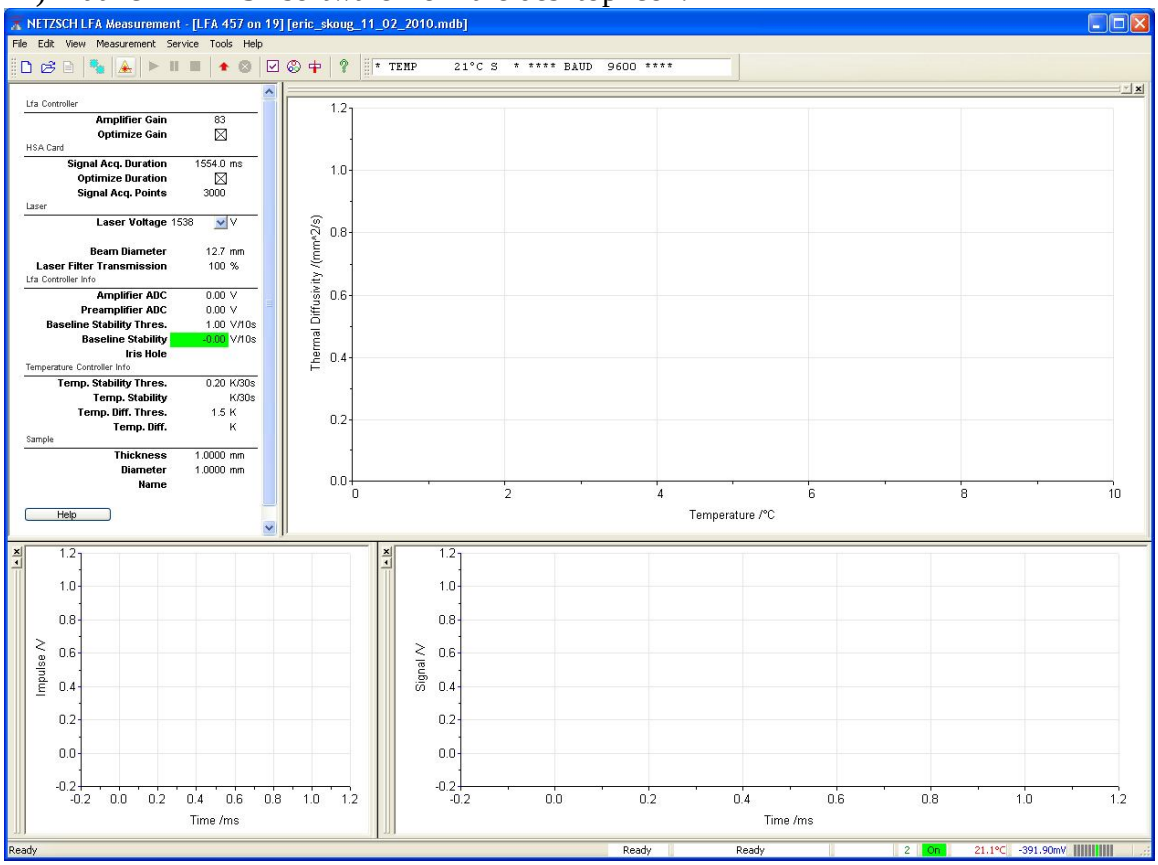


9) Rotate the furnace and detector assembly back into location, and close the furnace by simultaneously depressing the up arrow button on the front of the LFA-457, and the safety button on the right side of the system.
10) Check to assure the vent valve is closed.

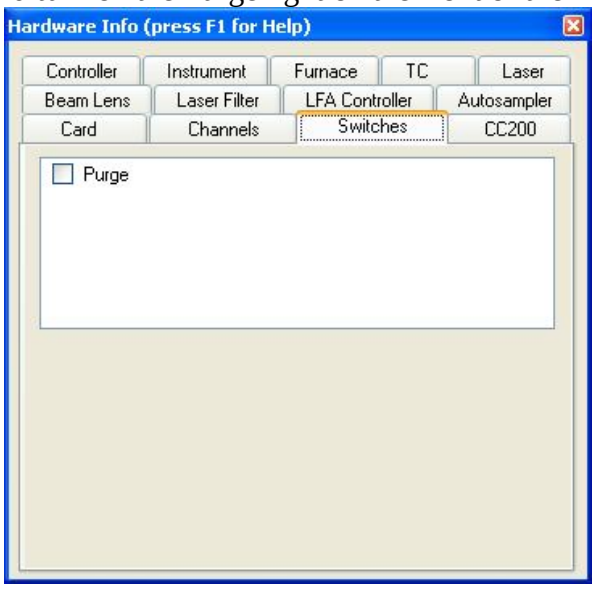
11) Turn on the vacuum pump
12) Slowly open the vacuum valve to prevent samples (or thermocouple) from being blown around, and pump to the lowest vacuum level as indicated on the front panel of the LFA-457.

13) Check that the regulator is open on the He/Ar cylinder and set to 5-10 psi. Close the vacuum valve, and slowly open the backfill valve to purge the sample space.
14) Repeat steps 12) and 13) for three backfills.
15) After the third backfill, the sample space should be at atmospheric pressure, and you can now turn off the vacuum pump.

16) Logon to the computer.
17) Launch LFA457 software from the desktop icon.



16) Select Service → Hardware Info → Switches and then click the box to turn on the purge. This should turn on the Purge light on the front of the LFA-457.



17) With the Purge light on, open the vent valve (red handle behind the detector container).

18) If this is the first experiment for the client then create a new database for the client, otherwise open the database for the client that is requesting the experiment.
19) In the software → upper left window, set the following initial values:

    LFA Controller section

        Amplifier gain = 40

    HAS Card

        Signal Acquisition Duration = 1000 ms

    Laser

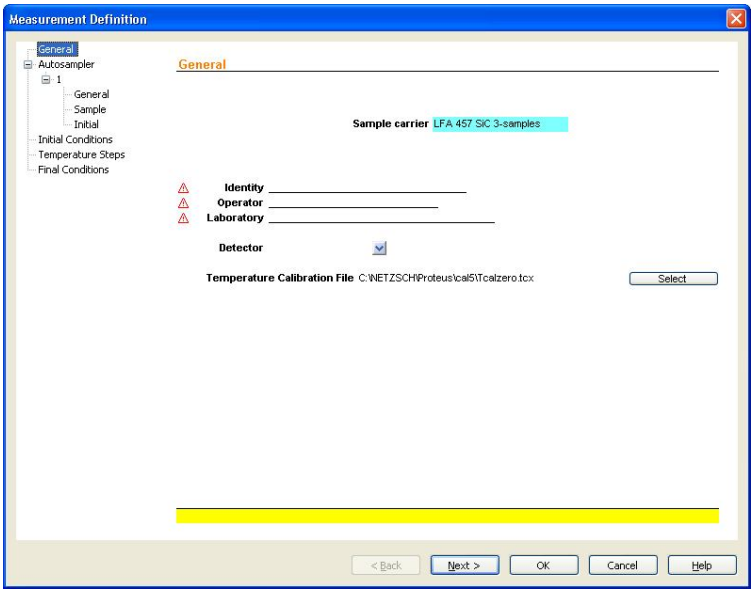
        Laser voltage = 1538 V
        Beam diameter = 12.7 mm
        Laser filter transmission = 100%

    Sample

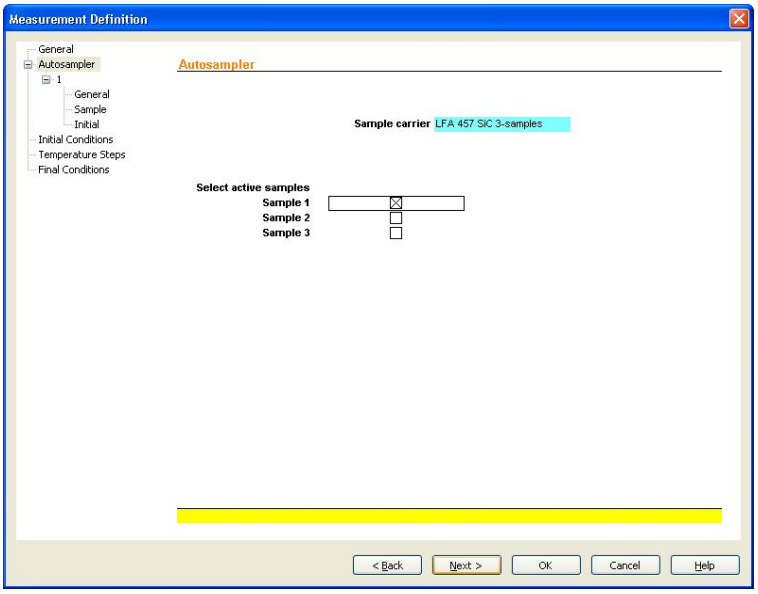
        Thickness = average value you obtained in the beginning
        Diameter = diameter of the sample

20) Select Service → Hardware Info → Autosampler and verify that the correct sample position is selected for the test shots.
21) Make a couple of test shots to find the correct Gain and Signal Acq Duration values and record these. Typical is to find a gain value that puts the curve in the 1 to 3 volt range. Signal Duration should be long enough to see the peak and the fall off.
22) Do this for each of the sample that will be processed.
23) Once all the values have been recorded go ahead and setup the full experiment. Select Measurement → New

24) The following should appear on the screen

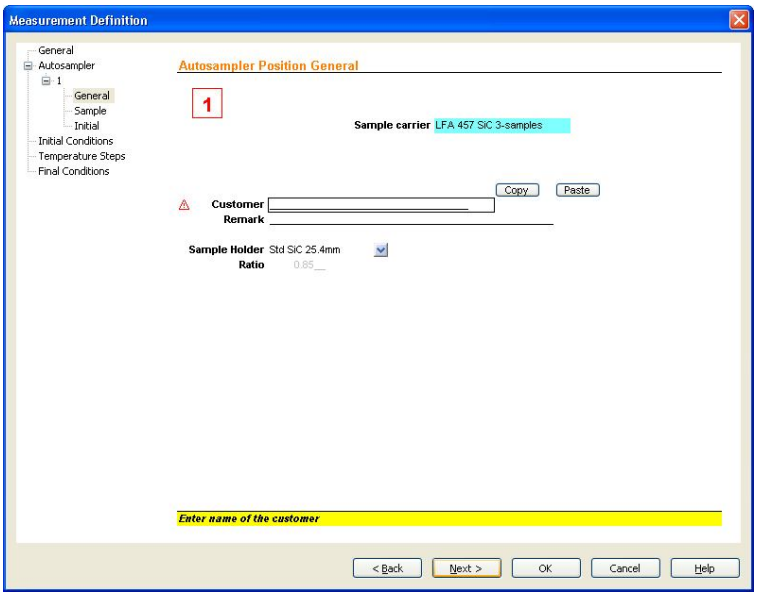


25) Fill in the Fields as follows
    1. Identity - should be the experiment number from the web site.
    2. Operator – should be your complete name
    3. Laboratory – should be ECE Testing Facility
    4. Detector – should be the detector install on the system
    5. Temp Cal File – should be left as the default
26) Select Next



27) Select the number of sample that will be measured

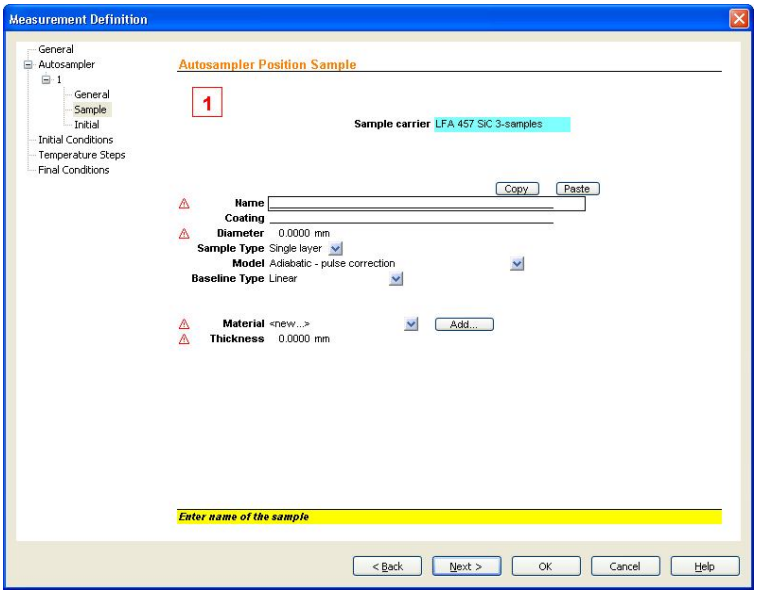
28) Select Next



29) Fill in the Fields as follows

1. Customer – will be the name of the person requesting the experiment
2. Remark – will be the experiment name from the website
3. Sample Holder – select the correct holder for the first sample

30) Select Next

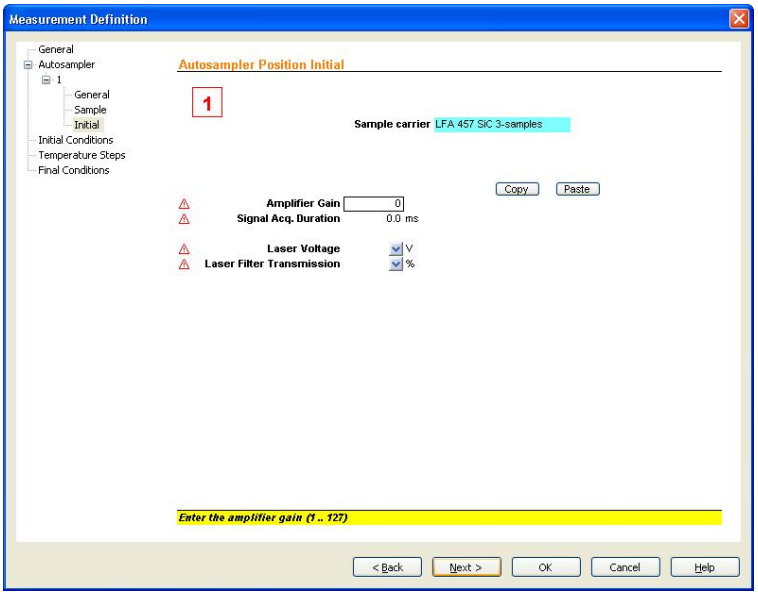


31) Fill in the Fields as follows

1. Name – is the sample name from the web site
2. Coating - should be Graphit
3. Diameter – should be the diameter of the sample being measured
4. Sample type – more than likely it is a single layer sample
5. Model – select Radiation + unless the correct model is known for material

6. Baseline – will be linear
7. Material – select the material being measured. If it is not in the drop down list then see Appendix A on how to add new materials.
8. Thickness – will be the average thickness of the sample.

32) Select Next

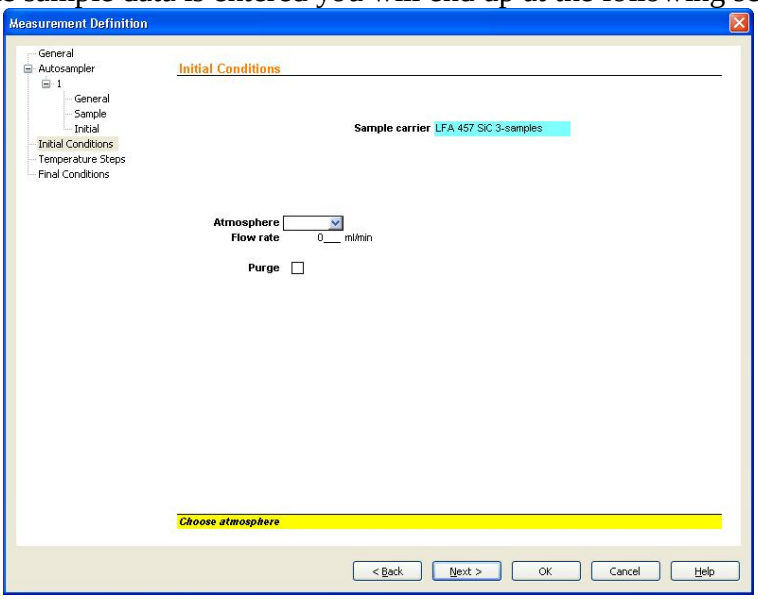


33) Fill in the Fields as follows
1. Amplifier Gain – will be the value from the test shot that best fits
2. Signal Acq. – will again be the best fit value from the test shots
3. Laser Voltage – more than likely will be 1538
4. Laser Filter – more than likely will be 100

34) Select Next

35) If you have more than one sample it will ask you for the information from bullet 28 again for each of the samples 2 and 3

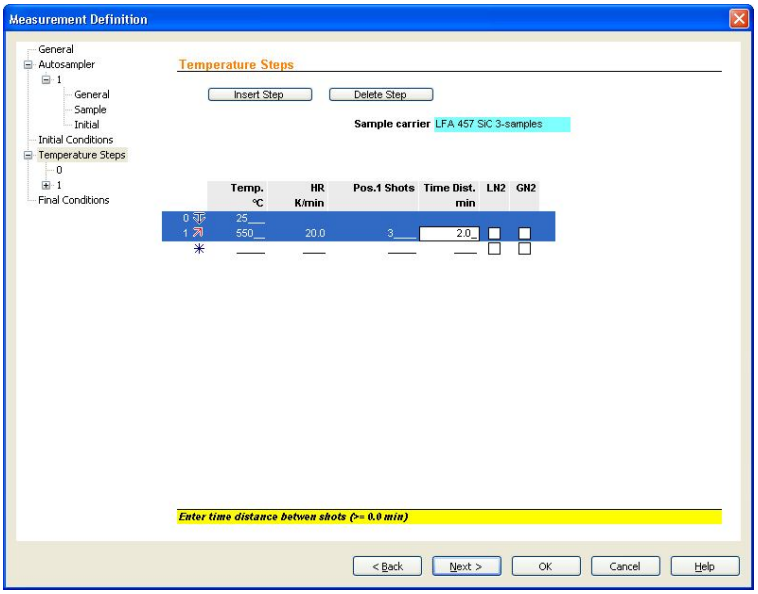
36) Once all the sample data is entered you will end up at the following screen



37) Fill in the Fields as follows

1. Atmosphere – will be the atmosphere the experiment is processed in
2. Flow rate – will be the flow rate of the purge during the experiment 80
3. Purge – check if purge is on during the experiment typically checked

38) Select Next

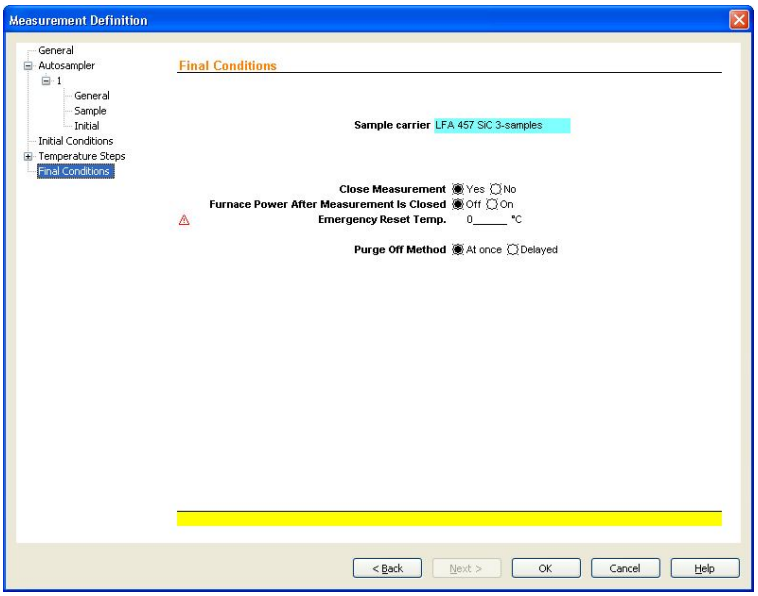


39) Fill in the Fields as follows

1. The first temp step will always be 25 since that is room temp
2. The second temp will be the first measurement point. If doing CP measurements with a standard you must make the temp changes in an ascending order. Otherwise, it works best to make all the measurements from the highest point in a descending order. So enter the value for the first temp step.
3. K/min – is the amount of temp change per minute between shots.
4. Pos. 1 Shot – how many shots at each temperature for that sample. If there is more than one sample there will be a value for each position.
5. Time Dist – is the wait time between shots. This is important if you have a material that retains the heat from the laser pulse. Typically 2 min
6. Continue this process until the correct temperature profile in entered to match the requested range from the web page.

40) Select Next

41) On the left hand side of the window select Final Conditions. The following window will appear



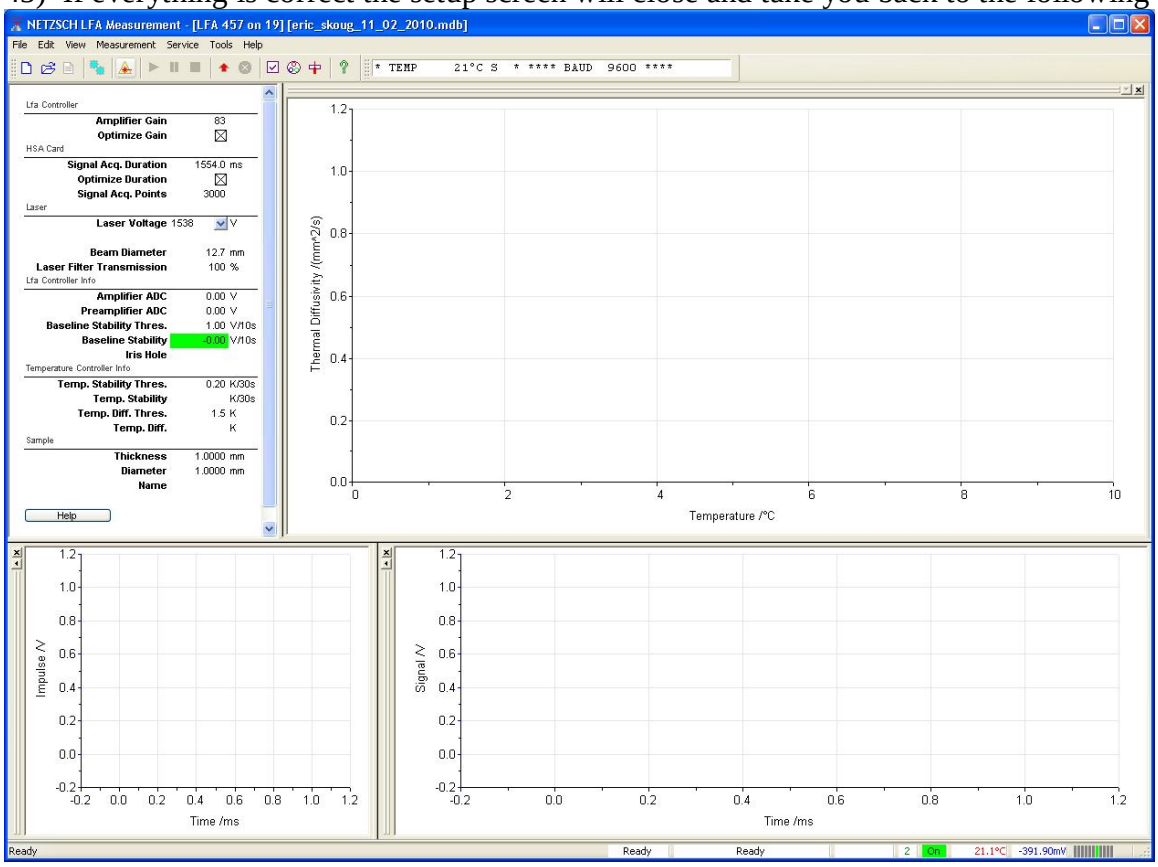
42) Set the following

1. Close the measurement – Yes, this stop the collection of data at the end of the experiment
2. Furnace Power – Off at the end of the experiment
3. Emergency Reset temp. – This value should be set to less than the melting point of the material. This is a safety feature to stop temperature run away.
4. Purge off – Typically 30 minutes after the experiment ends is a good selection.

43) Select OK

44) If there is an error anywhere in the profile the system will send you back to that point and ask you to correct the information.

45) If everything is correct the setup screen will close and take you back to the following



46) If everything is ready you can start the experiment by selecting Measurement -> Start

47) Depending on the number of measurement and the temperature range it may take 24 hrs for the experiment to complete. If the system is going to run for an extended period of time, the liquid nitrogen in the detector will need to be topped off about every 8-10 hours. If the detector runs out of liquid nitrogen cooling it will stop the experiment.

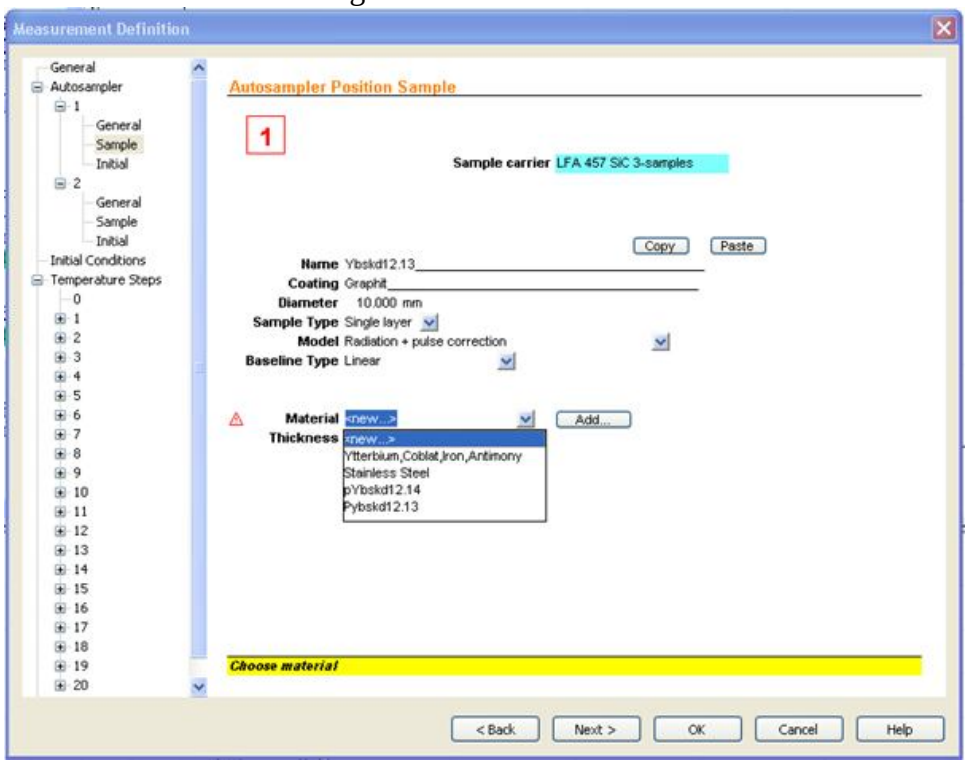
System Shutdown:

1) Collect the experiment data to be uploaded at the web page. Select the main experiment window and press Alt -> Prt Scr at the same time. Open WordPad and paste the experiment image in a new document Ctrl -> v. Save the WordPad document to the USB thumb drive. Copy the database in c:\Netzsch\proteus\data5\{database name} to the USB thumb drive.
2) Transfer the USB thumb drive to the laptop.
3) Open the WordPad document in Microsoft word and change the following
    1. Under Page Layout set the Orientation to landscape and set the Margins to Narrow.
    2. Right click on the image and select Format Picture
    3. Under Size set the width to 10.1 and select OK
    4. Print the file to the device doPDF. This will create a pdf image of the document. Save it to the default location which is the USB drive.
    5. Right click on the database file and select 7-zip -> Add to "{filename}".zip This will zip the database file and put a copy in the default folder on the USB thumb drive.
4) Complete the information about the experiment on the web site for the Netzsch LFA-457. Make a note of anything that went on during the experiment in the notes section. Upload both the screen shot pdf file and the zipped database file. If using the wireless network it may take a couple of minutes to upload the files.
5) Close the purge valve on the detector.
6) Open the furnace by depressing the button on the right side of the instrument and the down button on the front of the LFA-457 simultaneously. System will stop when it reaches full open. The furnace and detector assembly can then be rotated to the left (clockwise looking down) to get it out of the way.
7) Remove the samples from their holders. Put the samples on top of the system to be picked up by the end user. Remove the sample holders and put them in their storage location in the bench drawer.
8) Rotate the furnace and detector assembly back into location, and close the furnace by simultaneously depressing the up arrow button on the front of the LFA-457, and the safety button on the right side of the system.
9) Turn off the power to all the units. There is no order.
10) Close the main valve on the Ar/He tank
11) Experiment is complete.

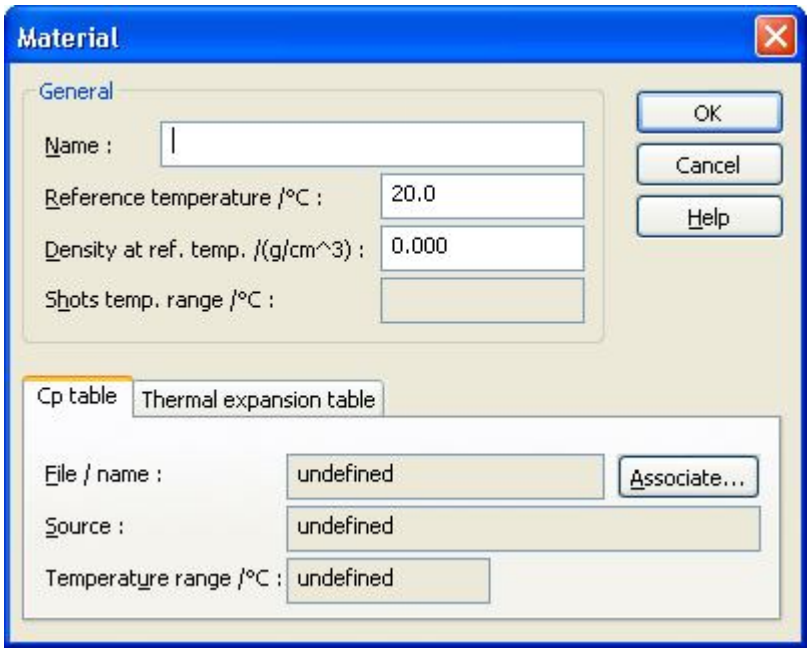
Appendix A:

Adding a new material to the software database

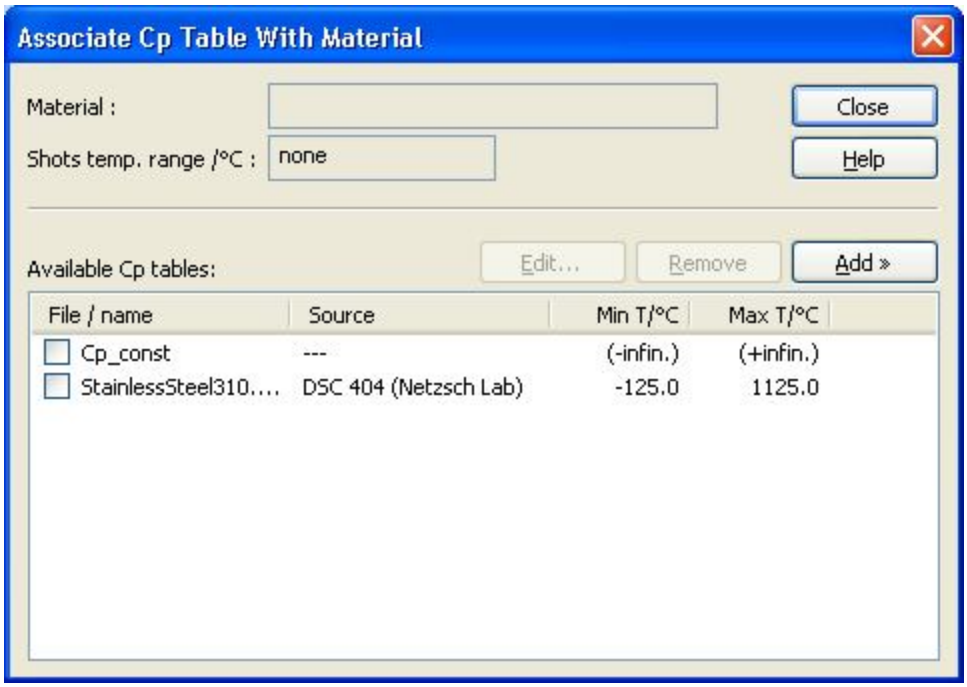
1) On the Measurement Definition screen you have the following information in select the material or adding a new material.



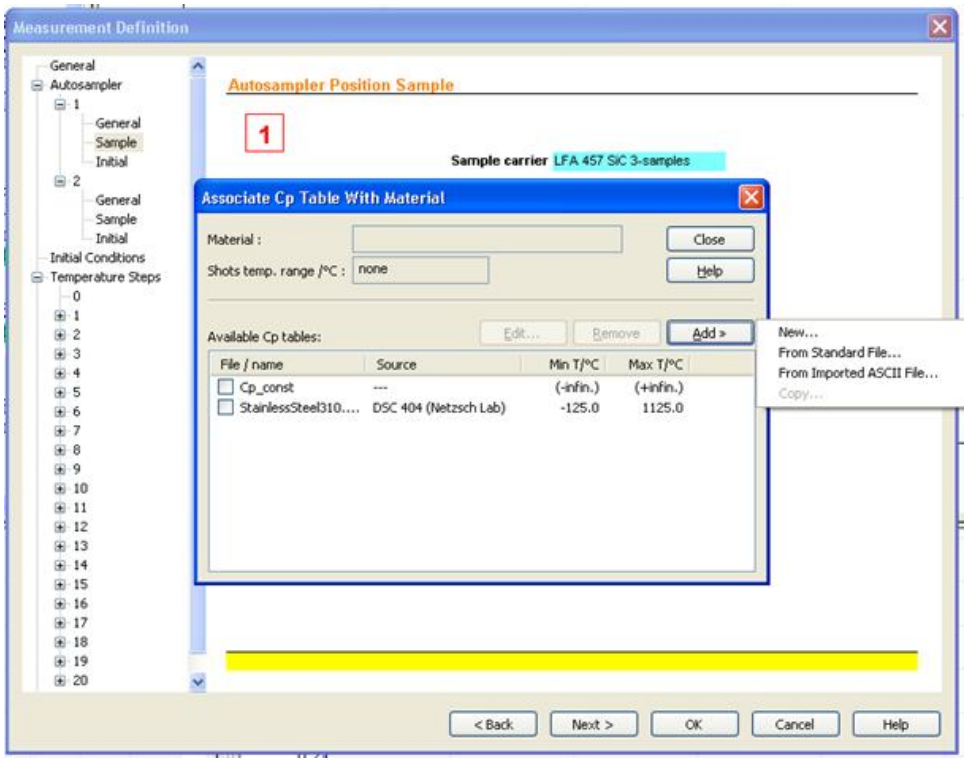
2) Select Add and fill in the known information



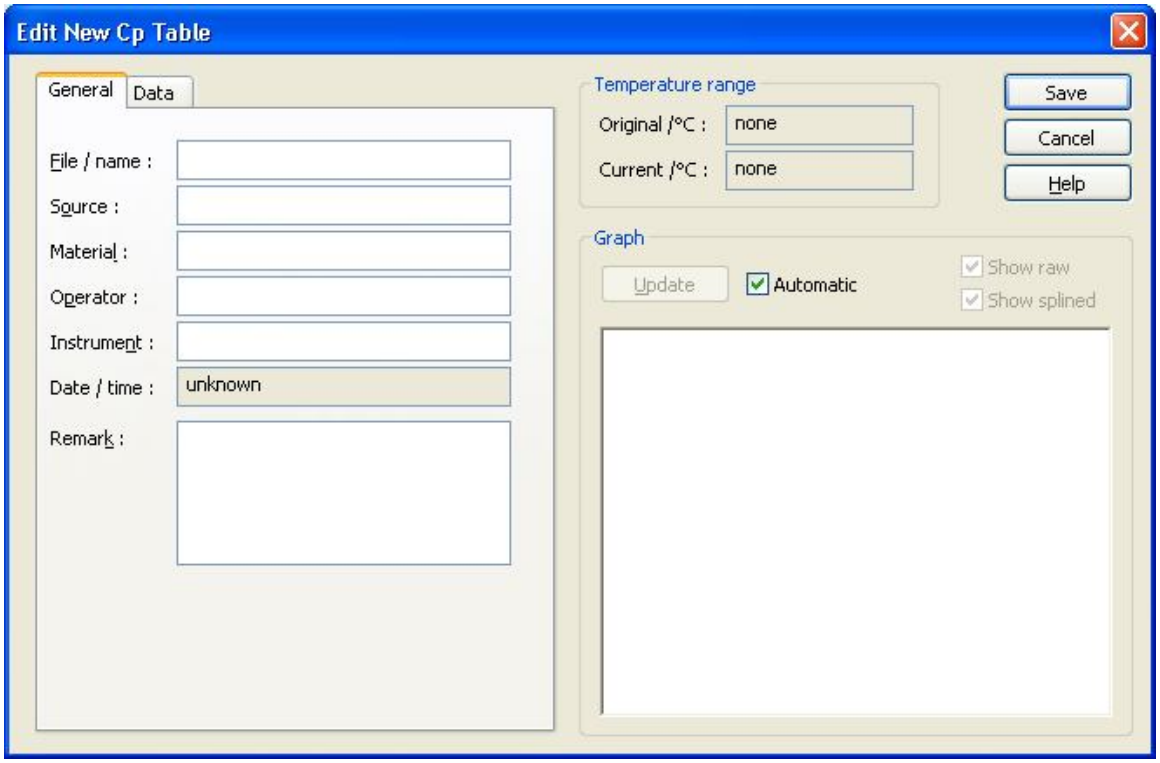
3) If you know the Cp table information select Associate. If the material is listed select it and close the window.



4) If the material is not listed you can select add and pick from New, From Standard Table or Import the Cp data from an ASCII file.



5) If adding the Cp data for a New material complete the following screen



6) The data can be entered under the Data tab. Select Save.
7) You will need to select the new Cp table data in the "Associate Cp Table With Material" window and select Close.
8) The same process can be followed for the Thermal expansion table tab.
9) Select Ok
10) Make sure to select the material in the Measurement Definition window.