BAXIT Differential Scanning Calorimeter

BXT-DSC-100

User Manual

DRAWELL
Artist of Science

Please read operating manual before installation and operation.

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CATALOG

1. Summary

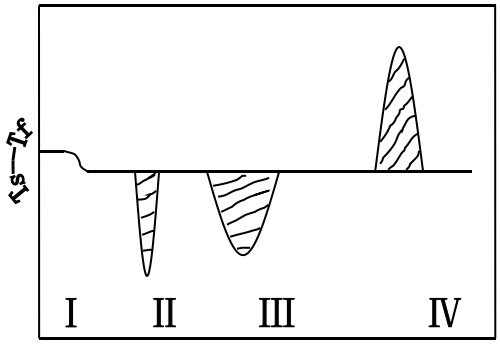
Differential scanning calorimetry (DSC) technology has been widely used. Differential scanning calorimetry is both a routine quality testing tool, but also a research tool.It measures temperature, heat flow relationship related to materials internal thermal transition.Our company's device is heat flux differential scanning calorimeter, with good repeatability, high accuracy characteristics, especially suitable for accurate measurement of the specific heat. The device is easy to calibrate, using a low melting point, fast and reliable, and with a wide range of applications, especially in materials research and development, performance testing and quality control. Characteristics of the material, such as the glass transition temperature, cold crystallization, phase transition, melting, crystallization, product stability, the curing / crosslinking, oxidation induction,etc.are research areas of differential scanning calorimeter.

Differential scanning calorimetry applications are: the curing reaction temperature and thermal effects of polymer materials, material phase change temperature and its thermal effects measurement, polymer materials crystallization, melting temperature and its thermal effects measurement, the glass transition temperature of the polymer material and so on. Subjects of experiment are: solid, liquid, viscous samples, except the gas.

Place the specimen and the reference material into the crucibles respectively, heating in the oven to change the temperature of the specimen and the reference material. If hot melt of the specimen as same as the reference material and the specimen has not the thermal effect, the temperature difference between the two is close to "0", then we can get a smooth curve. As the temperature increases, the specimen produces a thermal

effect, but the reference material doesn't produce the thermal effect, it makes the temperature difference between the two, it expresses as the peak in the DTA curve, the bigger temperature difference, the greater the peak, and the more the change number of temperature difference, the more the number of peaks. It is called peak exothermic which peak upward, and it is called peak endothermic which peak downward.

The picture is a typical DSC curve; it shows four types of changes.



Typical DSC Curve Temperature coefficient→

Ⅰ is second order transition; it is the change of level base line.

Ⅱ is the endothermic peak, it caused by the melting changes of the specimen.

Ⅲ is the endothermic peak, it caused by the decomposition reaction of specimen.

Ⅳ is the exothermic peak, it caused by the crystalline phase transition of the specimen.

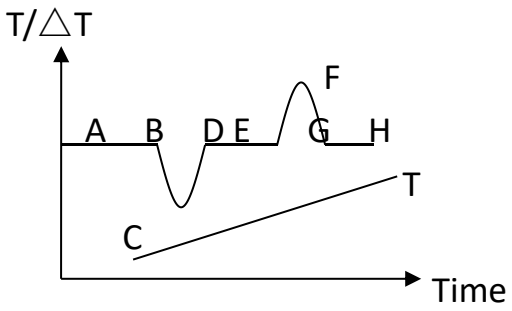
2. Experimental principle

Materials are often accompanied by thermal effects in the process of physical changes and chemical changes, exothermic and endothermic

phenomena reflect the heat enthalpy of the material has changed. DTA can measure the functional relationship of temperature difference between the specimen and the reference material to temperature or time in the same heating conditions.

Differential scanning calorimetry is a technique,which in the case of process control temperature, measures the relationship of power difference and temperature between the output material and reference material. Our company's device is heat flux differential scanning calorimeter, the ordinate is heat flow difference of the sample and the reference material, in units of mw. Abscissa is the time (t) or temperature (T), from left to right for the growth (does not meet this requirement should be specified).

After putting the specimen and the reference material into crucible, according to the heating rate, if the hot melt of the reference material similar as the specimen, we can get the ideal scanning calorimetric analysis diagram.



In this picture, T reflects the temperature curve of thermocouple which sticks in the reference material; Line AH reflects the temperature difference curve between the specimen and the reference material. If the specimen without thermal effects, △T between the specimen and the reference material is 0, line AB, DE, GH is smooth baseline in the curve. When the thermal effect occurs, if the temperature of specimen lowers than the temperature of reference material, we can get the endothermic peak like line BCD; otherwise, we can get the exothermic peak like line EFG.

The number, position, peak area, direction, height, width, symmetry of the peaks reflect the times of physical changes and chemical changes in the measured temperature range, temperature range which changed, the size , positive and negative of thermal effect . The heights, width, symmetry of the peaks are not only relating with test conditions, but also relating with pharmacokinetics factors in the process of the specimen change, the result is much more complex than the ideal curve.

3. Instrument features

1. New furnace structure, better resolution and baseline stability;

2. Digital gas mass flow meter, precise control of the purge gas flow; Data is directly recorded in the database;

3. The instrument can be bi-directional control (host control, software control); friendly interface, easy to operate.

DSC range	0 ~ ±500mW
Temperature range	Room temperature~600℃
Heating rate	0.1~80℃/min
Temperature resolution	0.01℃
Temperature repeatability	±0.01℃
DSC noise	0.001mW

DSC resolution	0.001mW
DSC accuracy	0.001mW
DSC sensitivity	0.001mW
Temperature control method	heating, constant temperature (full program automatic control)
Curve scan	heating scan
Atmosphere control	automatic instrument switching
Gas flow rate	0-200mL/min
Gas pressure	0.2MPa
Display mode	24bit color 7-inch LCD touch screen display
Data interface	standard USB interface
Parameter standard	Equipped with standard materials, with one-key calibration function, users can calibrate temperature and enthalpy by themselves
Power supply	AC 220V 50Hz or customized
Power	600W

4. Interface Operation

4.1. Start up and stop interface

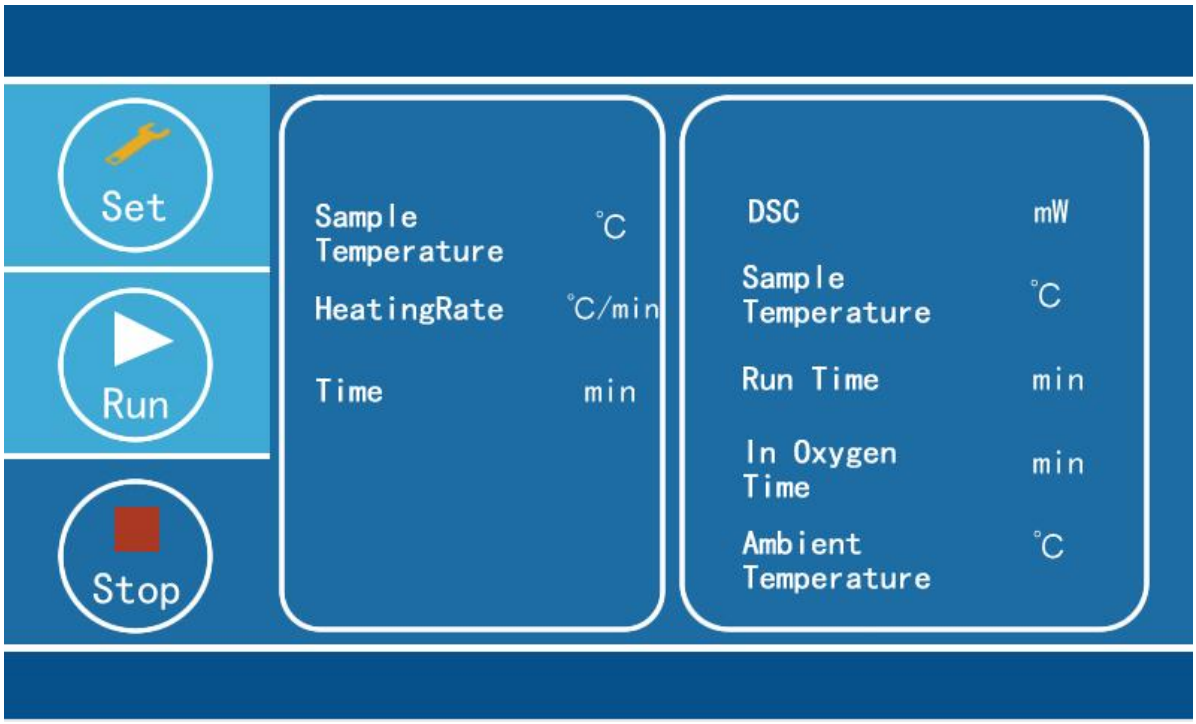


Figure 1.1

a. Click  button to enter into set interface to get the temperature of specimen, heating rate, gas flow.

b. Click  button, to start the system.

c. Click  button, to enter into temperature adjustment interface.

4.2. Set Interface

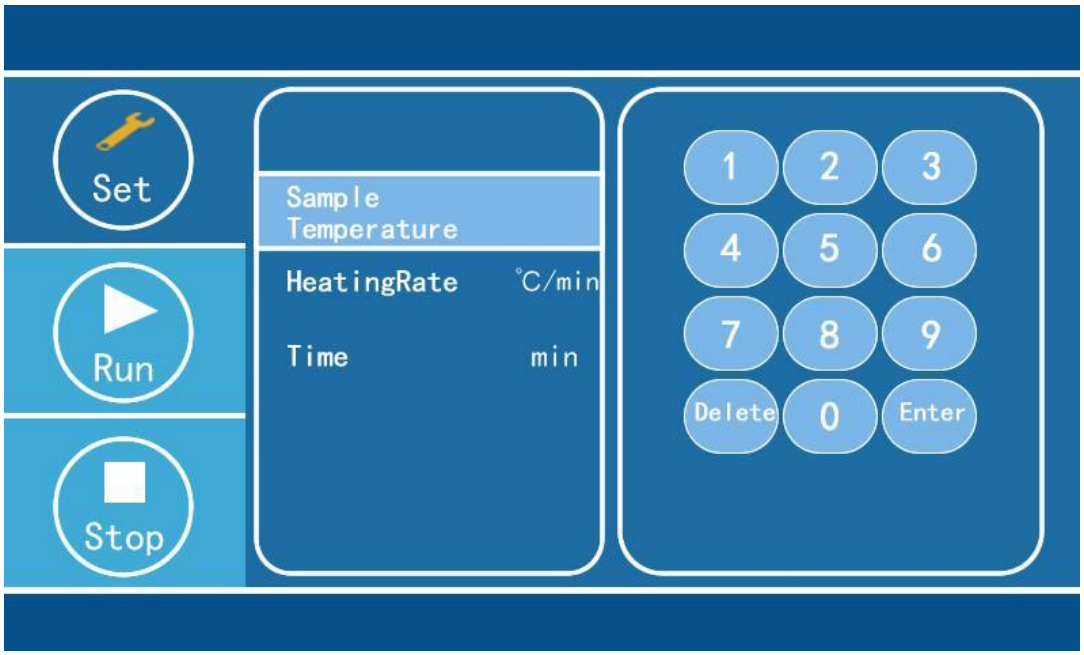


Figure 1.2

a. Click  to enter into the interface for setting specimen temperature, click number key, input the temperature of specimen which is needed to set.

b. Click  to end the set of specimen temperature.

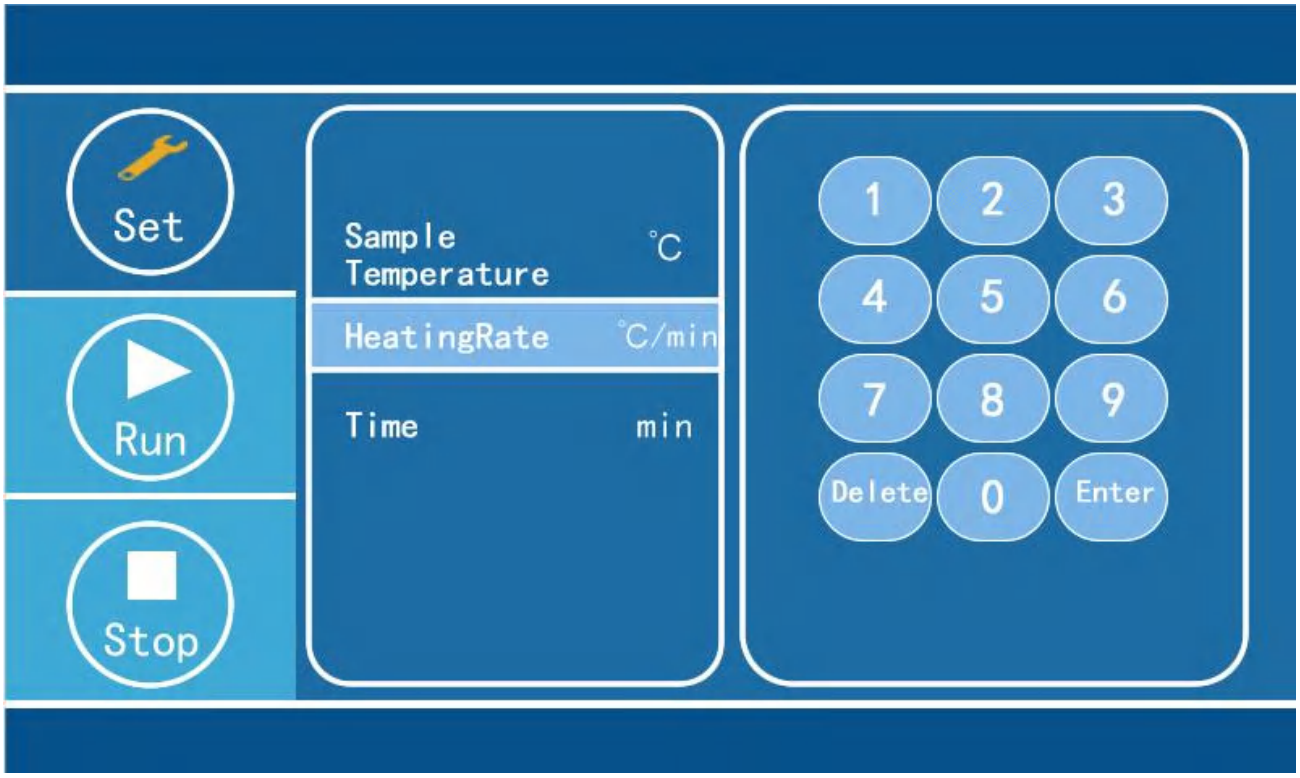


Figure 1.3

c. Click  to enter into the interface for setting heating rate, click number key, input the heating rate which is needed to set.

d. Click  to end the set of heating rate.

4.3. Run interface

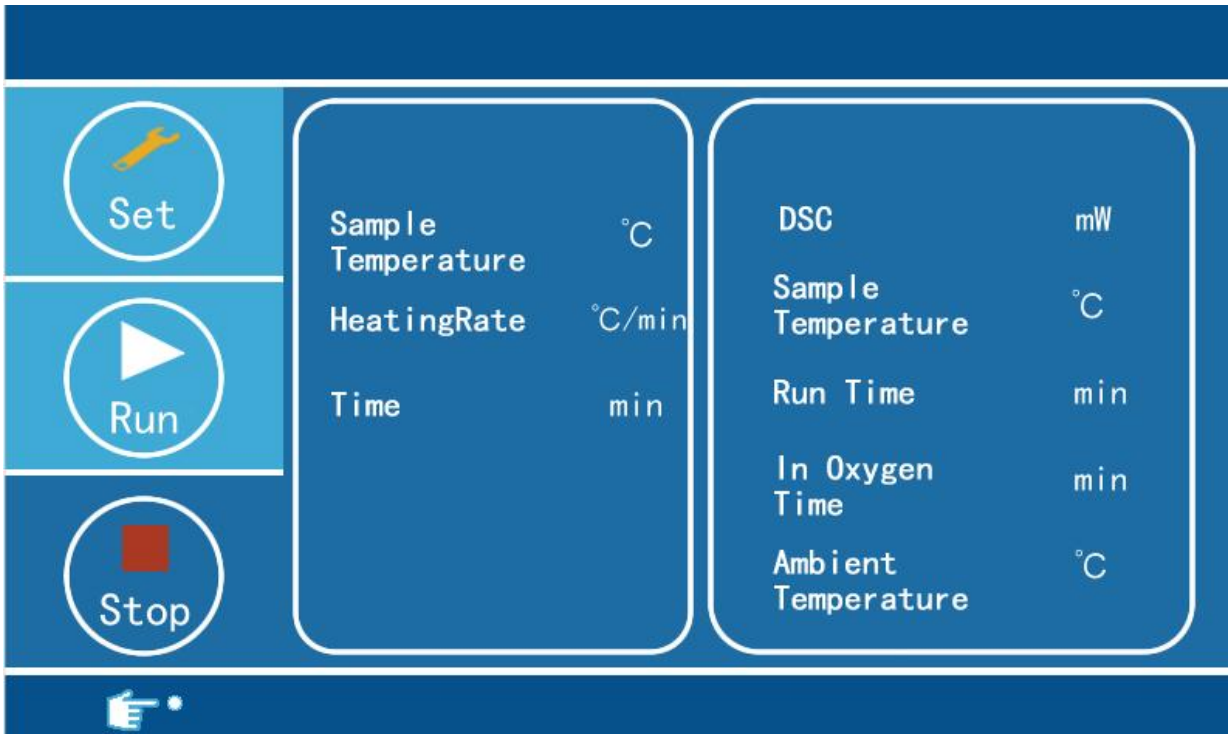


Figure 1.4

a. Click  to stop the system.

b. Unable to set the instrument in runtime.

4.4. Temperature adjustment interface

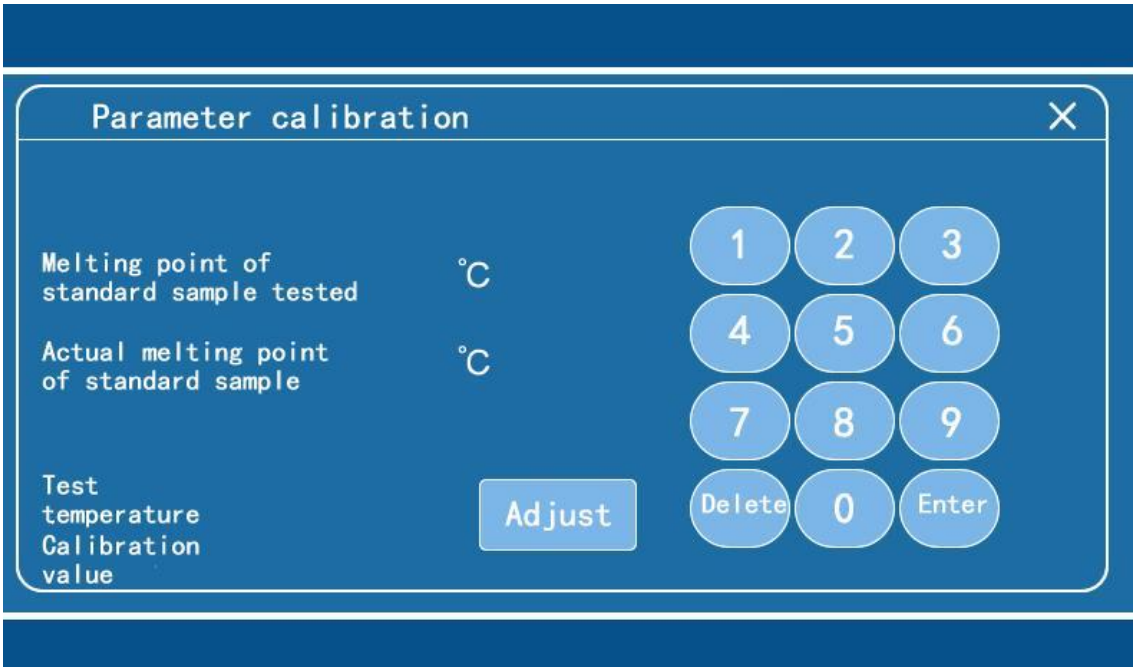
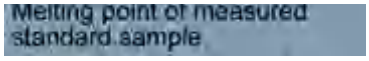


Figure 1.5

a. Click temperature value to turn into white in black, click the number key, input temperature.

b.  is theoretical value of the standard material, and  is the actual test values.

c. Click  after input, close the power of instrument, and switch on, calibration finished.

d. If there is no modification or adjustment, click  to close the dialog box.

Settings drop-down menu "X-Temp"

5. Description of software

5.1. Software interface

1. Click on the software icon into the startup interface （As shown in Figure 1-1）

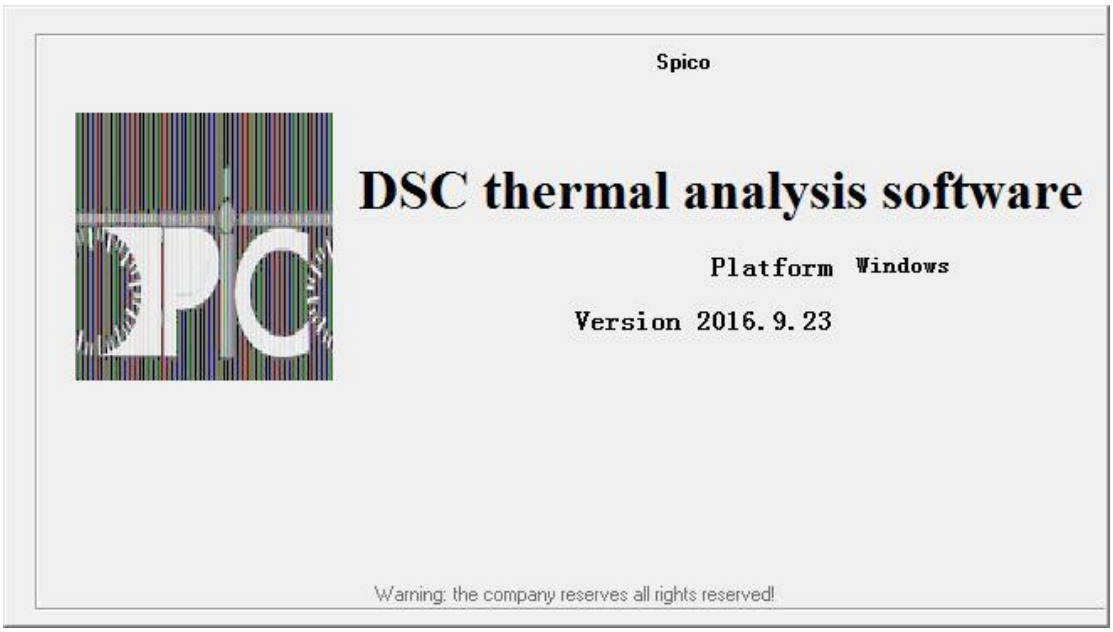


图 1-1

After entering the start interface, press 【**any key**】to enter the main interface。If the keyboard does not respond to the software to stay in the interface.

2. Parameter setting of the main interface（As shown in Figure 1-2）

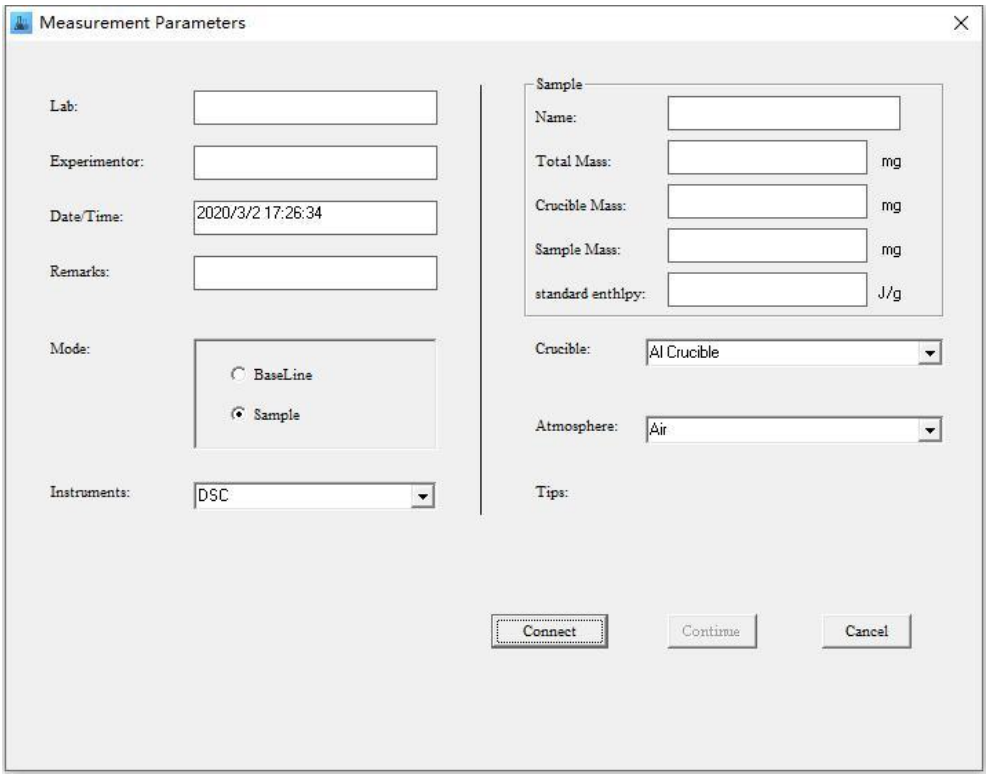


图 1-2

Press the【Preferences】 button in the parameter setting interface to carry on the common experiment parameter setting.

【Plot】button on the main interface（As shown in Figure 1-3）

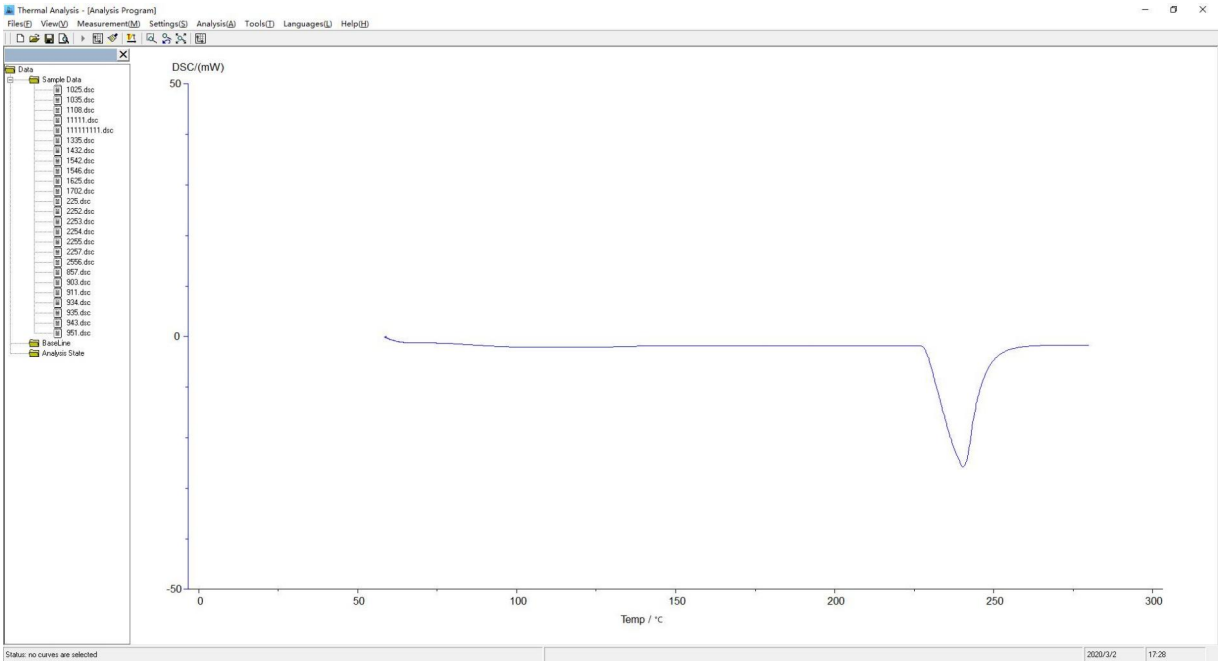


图 1-3

Click on the【**Plot**】button to enter the drawing interface

5.2. Direction for use

a.Communication link

Open the software to enter the startup interface, press 【**any key**】to enter the main interface of the software. Enter the parameters to set up the main interface, fill in the experimental information and set the experimental parameters. Then click the 【**Connect**】 button in the 【**Settings**】menu **(S)** to connect with the PC software. If the communication connection is successful, the parameter setting interface will display the 【 **Device connected successfully**】.

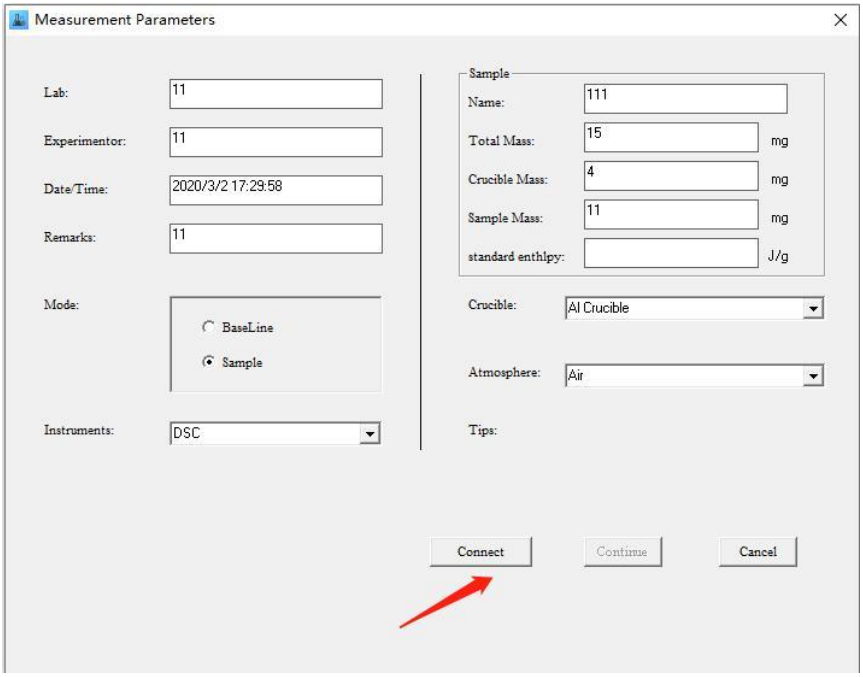


图 1-4

b.Temperature control program

After successful connection, the device should be set up in the temperature control program. The first time you click the【**add temperature section**】button will pop up the【 **initial temperature**】 input window, and then will pop up to add temperature window (The starting temperature of 24 C is the ambient temperature of the experimental instrument.) After setting the temperature control program, click on the 【**Transfer**】button to complete the temperature control program.

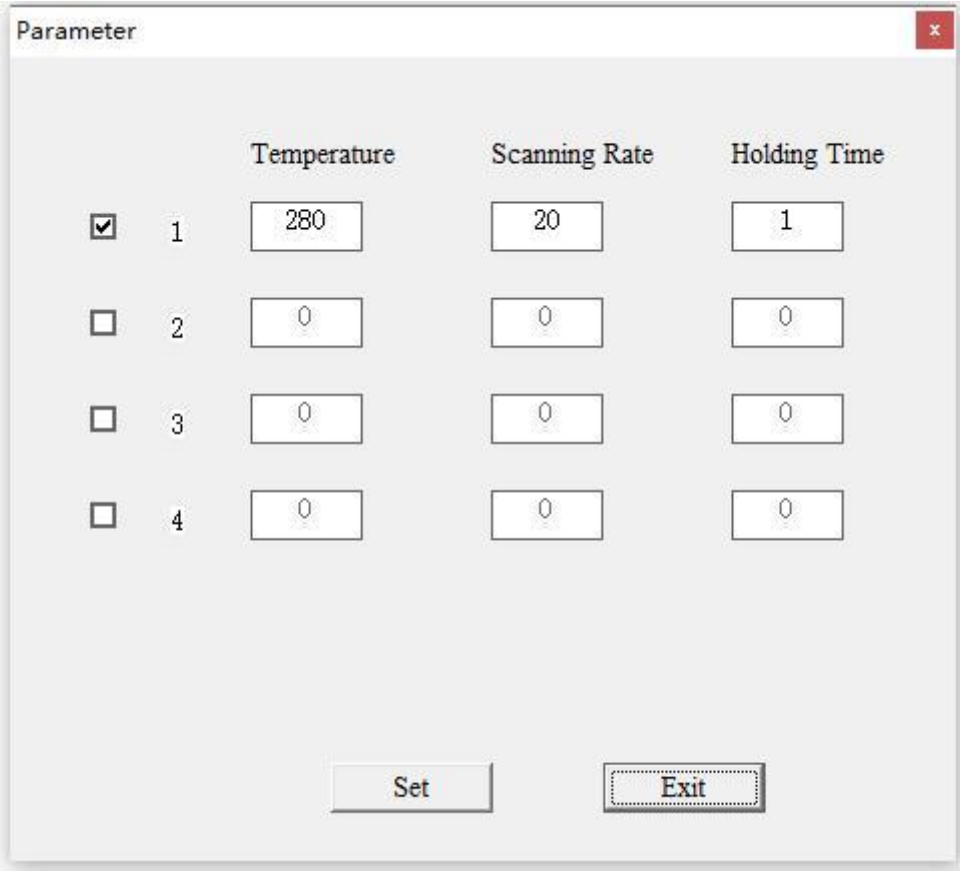


图 1-5

c.art experiment

After the success of the temperature control program, click on the 【plot】(F) menu under the 【start】 (S) button, the software began to receive automatic data graphics. Before clicking the start (S) button, the drawing interface can be switched to the "X-Time" interface or the "X-Temperature" interface according to the parameters of the interface coordinate format. X-Time "interface is used to measure the oxidation induced phase。 Click to set the drop-down menu "X-Temperature" to open another interface, which is used to measure the melting point (enthalpy), glass transition temperature, instrumental coefficient and crystallinity. According to the experiment, it is needed to choose one of the experiments at will.

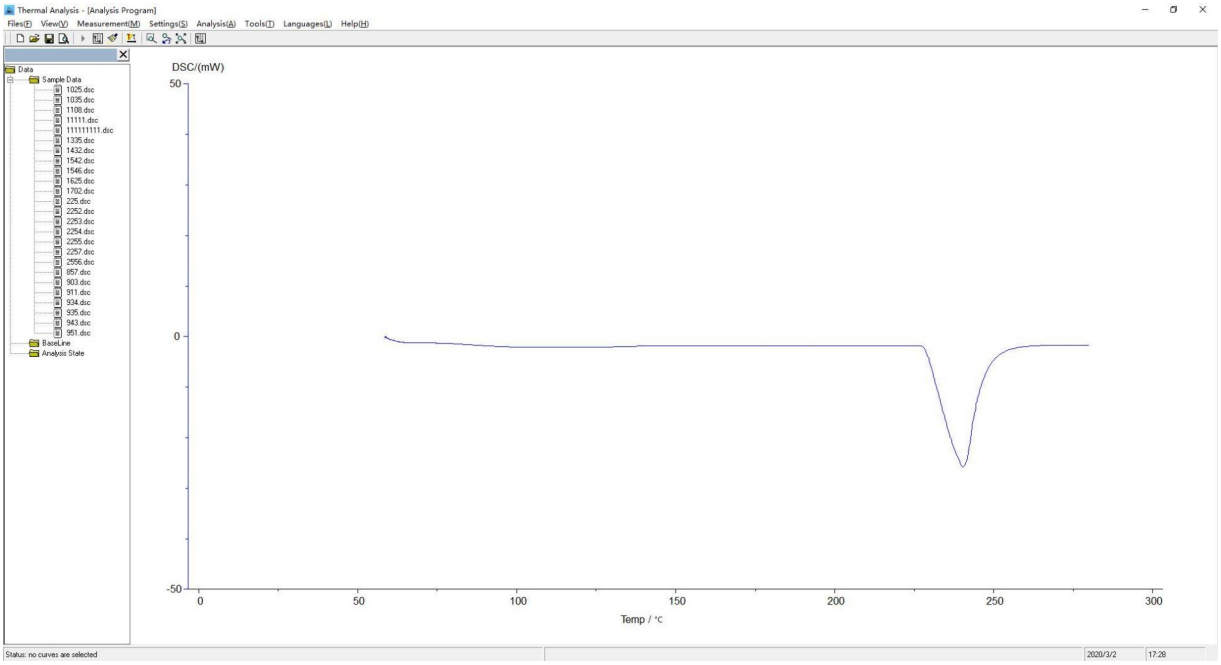


图 1-6

d.Software detail description

When the software receives the real-time data, it will pop up the real-time data window to display the real-time change of the data dynamically. When the real time data window is docked at the top of the screen, the mouse is moved out of the window, the window will automatically pull hidden. When the mouse moved to the vicinity of the window will automatically drop the display. If you don't hide the real time data window, just drag the window to the top of the window. If you close the real time data window, click the real time data button of the window menu which will pop up the window again.

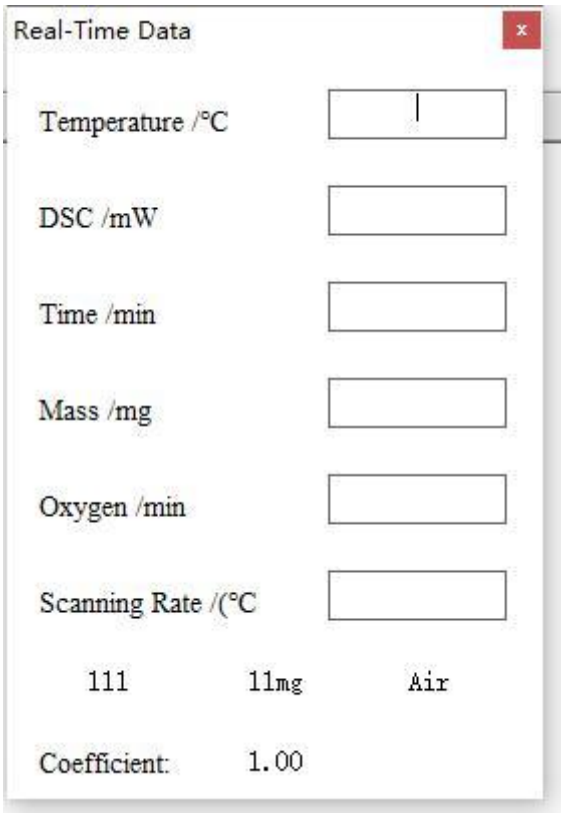


图 1-7

only the value of the instrument coefficient in real time data window can be directly filled.

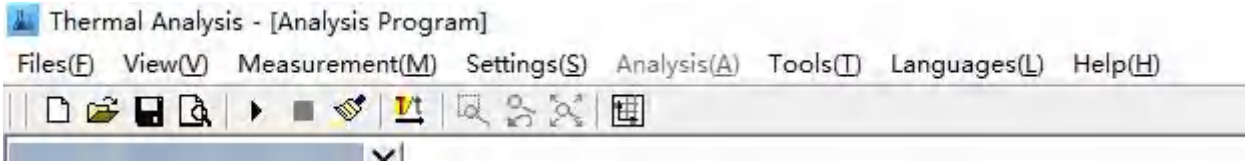


图 1-8

Correspond to the following functions:

Open Save Print Generate Began Stop Clear the screen Image Help Exit Reports

e.Experimental data processing

The oxidation induction period, the calculation of enthalpy and the glass operation are in agreement with the previous DSC software. In the drawing interface, click the right button will pop the following shortcut menu.

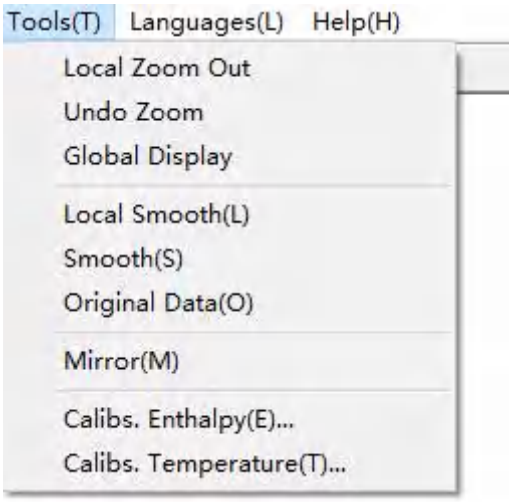


图 1-9

f.Movement of curves and annotation of drag label

Curve movement

Because the temperature control program can be set up to four, so the curve of the experimental data is also up to four. Under the X-Temp interface, the four curves were red, blue, green and purple.

If you want to move a curve just right pop-up shortcut menu, and then click on the curve of the mobile button to select the corresponding color options, and then press d the key to drag the gear.

After the completion of the curve movement, click Cancel button to cancel the curve movement.

Drag label

Marked information is divided into two steps. The first step is to drag and drop the label information of the selected curve. The second step is to move the mouse arrow to the tail of the label drag and drop. And then click on the mouse ,when the arrow is removed. At this point the mouse arrow is changed from the cross into the four dimensional shape. At this point, the information can be marked with the key gear drag and drop.

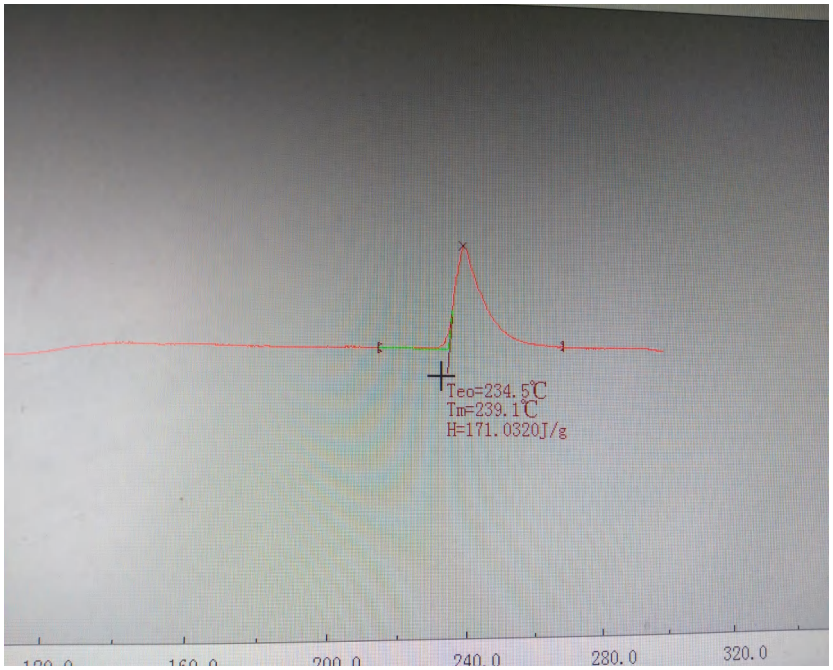


图 1-10

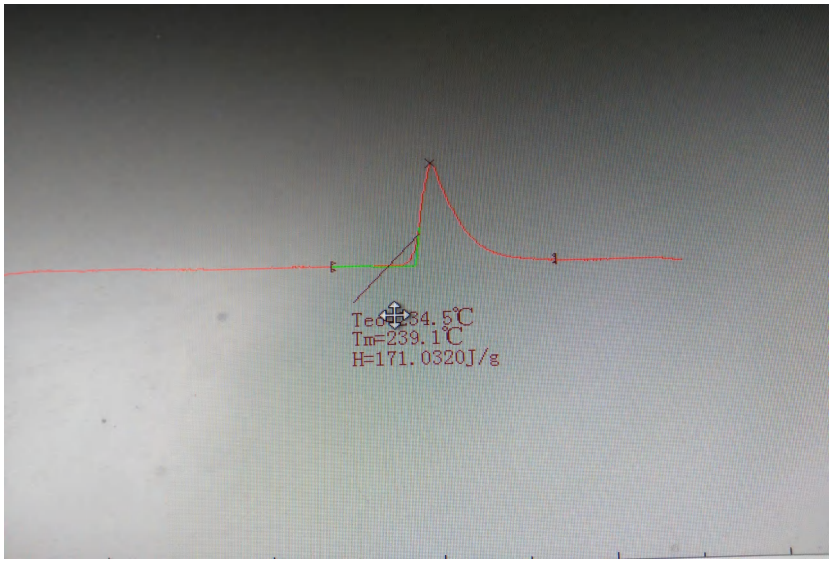


图 1-11

Click the Cancel button to cancel the drag and drop function when the label information is finished.

6. Instrument Calibration

6.1. Temperature correction

Purpose:

The instrument indicates that the temperature is equal to the true temperature of the sample.

Measuring the phase transition temperature of standard substance in

accordance with the sample measurement method, generally use tin, indium, zinc standard material correction, you can also choose other standard material upon request.

Procedure:

1. Turn on your computer, and connect the instrument data cable to the computer, plug in the power to the instrument, turn the instrument on the back of the switch.

2. Open the software, click 【 Settings 】 in the menu bar, click 【Communication Connection】, when showing the connection is successful, that means the instrument is connected to the computer.

3. Initial screen is oxidation induction period test interface, click Coordinate Selection X-Temp in the【Settings】to another interface.

4. In the 【Setup】 option, select 【Parameter Settings】, the dialog box shown in Figure 3.1. The max temperature is set to 350 ℃. Heating rate is set to 20 ℃ / min, holding time is set to 0min.

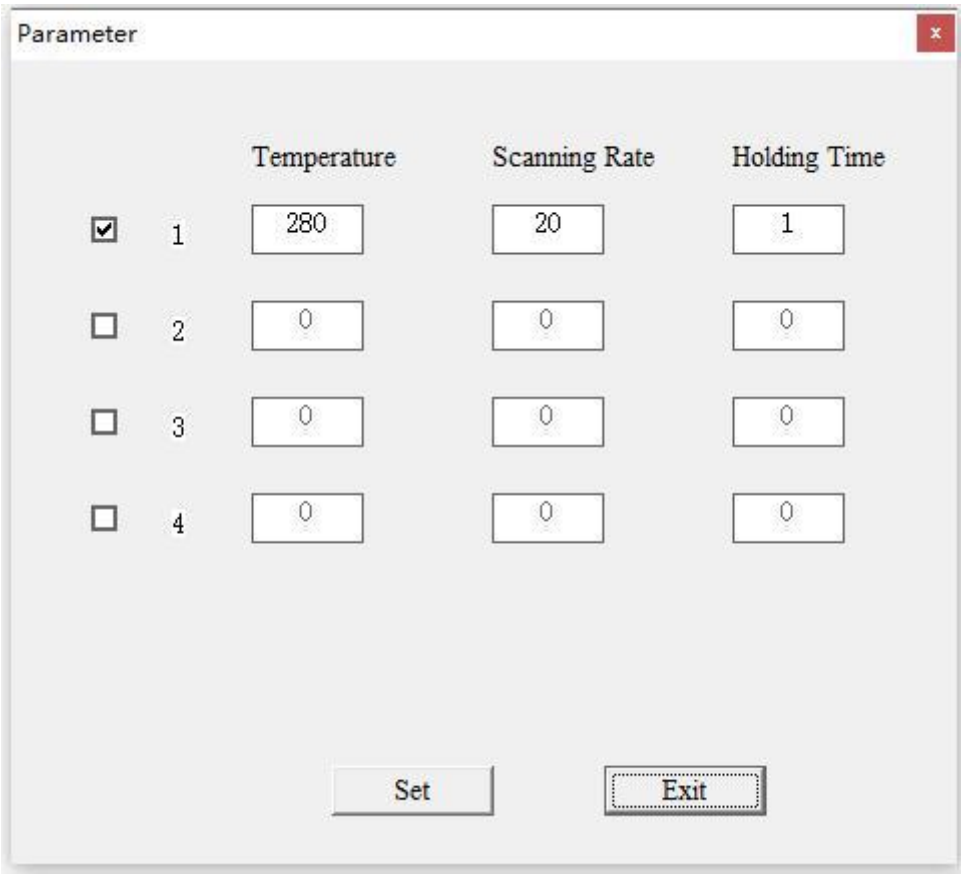


Figure 3.1

5. Take delivery of tin particles in the aluminum crucible, with tweezers put the aluminum crucible with tin particles in it into the center of the sample tray, and take another empty aluminum crucible as a reference. Close the lid.

6. Click the button in shortcut menu, and start the experiment.

7. After the DSC curves shows a full peak, you can click button on the shortcut menu, and stop the experiment.

8. Click on the menu bar【Data Analysis】, select【curve smoothing】, the system can automatically correct curve. Click【Data Analysis】, select 【Melting point (enthalpy)】, the dialog box shown in Figure 3.2, click OK. Before the change in the curve starts, left click, right-click when the curve ends the change, the dialog box shown in Figure 3.3, Click "No", i.e. in the figure shows extrapolated onset melting temperature Teo of tin. If the selected starting point

or ending point is incorrect, you can click "Yes" to re-select when shows the dialog in Figure 3.3.

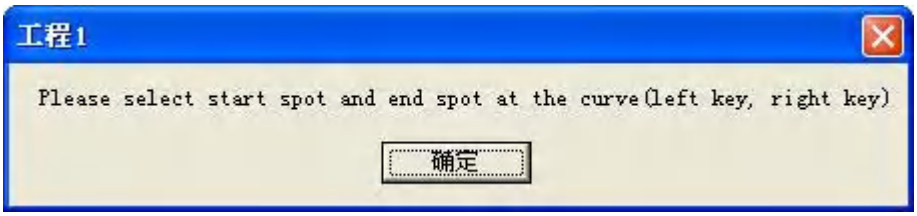
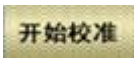


Figure 3.2

Figure 3.3

9. The actual melting point of tin is 231.9 ℃, experiment measured Teo is not within the range of 231.9 ± 1 ℃, click on the left bottom corner, click  , the interface shown in Figure 1.6, enter 231.9 in【Standard sample measured melting point】and enter actual measured melting point value in【Actual melting point】, press【OK】, then click 

, turn off the instrument calibration interface.

10. After calibration, the first to shut down software, and then turn the instrument off, then re-open the software, instruments, measure again after a successful connection tin melting point values, if the actual measured temperature is not in the range of 231.9 ± 1 ℃, repeat the above operation until tin melting point values in the range of 231.9 ± 1 ℃.

6.2. Calibration objects choice

Do occasional correction for temperature to ensure test accuracy. According to the sample actual test temperature, select the calibration object. Calibration object selection principle: the extrapolation calibration object temperature and the temperature of the sample test project to be measured should be relatively close to ensure the accuracy of the test. For example:

A. The test sample is an epoxy-based polymer material: (glass transition temperature Tg of 60 ℃ --- 140 ℃), benzoic acid used: temperature Teo of extrapolation is 121.0C-123.0 ℃

B. The test sample is an epoxy, polyester powder coating material: (glass transition temperature Tg of less than 70 ℃), benzophenone used: extrapolated temperature is 38.6 ℃.

C. The test sample is solder substances, use of pure tin: Teo extrapolated temperature was 231.9 ℃

The following table is commonly used calibration object melting point and theoretical enthalpy values:

Standard Substance	Theoretical melting point ℃	Theoretical melting enthalpy J / g
In	156.6	28.6
Xi	231.9	60.5
Zn	419.5	107.5

7. Instrument application

7.1 Melting point (enthalpy) measurement

Melting point is transition temperature of the material from the liquid crystal phase; the thermal analysis measured the most common one physical data. The measurement accuracy and thermodynamic equilibrium temperature error is up to ± 1 ℃ or so. ICTA recommended method is currently used to measure the melting of a solid material endothermic peak. Figure 4.1 corresponds to point B in Figure B' is the starting temperature Ti, G point is the temperature corresponding to the extrapolated onset temperature Teo, the leading edge of the peak maximum slope of the tangent line of the previous intersection of the baseline extension, C point the temperature corresponding to the temperature of bee top Tm, D corresponding to the point D' is the end temperature Tf.

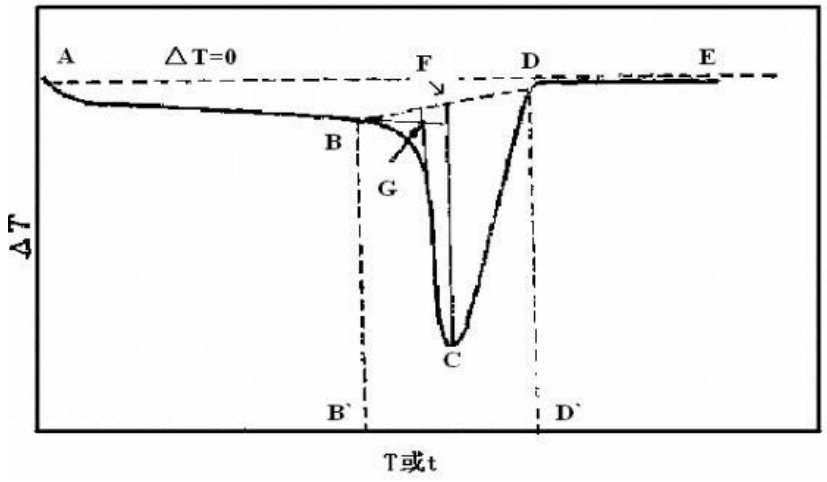


Figure 4.1

Enthalpy is a state of matter system energy function, which is numerically equal to the system with the internal energy U pressure P and the volume V of the product, i.e. H = U + PV. Under certain conditions and the environment from the system to measure the heat transfer between the system and the enthalpy change of the internal energy values. The condition if no other power system under isochoric process in all the heat absorbed to increase the internal energy

of the system in the isobaric heat absorbed in the process, all for the enthalpy increase, due to the general chemical reactions are in such pressures are carried out, so the enthalpy of more practical value. The DSC curve can be obtained by calculating the peak area of the sample melting enthalpy, i.e. the figure BCD.

Procedure:

1. First, open the purge gas, the gas flow control about 120ml/min. Atmosphere is generally used as nitrogen, helium, argon and other inert gases, some specimens can also be carried out in an air atmosphere.

2. Turn on your computer, the instrument data cable connected to the computer, plug in the power to the instrument, and turn the instrument on the back of the switch.

3. Open the software, initial interface is oxidation induction period test interface, turn to the melting point (enthalpy) measurement interface.

4. Click 【Setup】 option in the menu bar and click 【communication connection】to show the connection is successful, the instrument connected to the computer.

5. In the 【Setup] option, select Parameter Settings】, the dialog box shown in Figure 3.1. The ending temperature parameters should be slightly higher than to be measured sample temperature. If the desired temperature range of the sample is unknown, the ending temperature can be set a little higher, generally 550 ℃, the heating rate is typically set to 10 ℃ / min, constant time 0min, can also be determined according to the specific operational requirements.

6. As needed to prepare a reference sample, an empty crucible generally used to make the reference.

7. Weighed amount of sample, usually 10 ~ 20mg, placed in a crucible.

8. Open the DSC furnace (guaranteed at room temperature); with

tweezers put the aluminum crucible with tin particles in it into the center of the sample tray, and take another empty aluminum crucible as a reference. Close the lid.

9. In the experimental data field in the interface, input 【quality of the sample】, in milligrams. Click the  button on shortcut menu, the instrument starts the experiment.

10. This measure is the melting point of the sample, as a complete DSC curve peak appears, you can click the  button on the shortcut menu, stops the experiment. After the end of the experiment, on the shortcut menu click  button to save first the test data to avoid data loss.

11. While processing data, click  on the shortcut menu button, open curve data need to be processed, click on the menu bar【Data Analysis】, select【curve smoothing】, the system can automatically correct curve.

12. Click on the menu bar【Data Analysis】, select【curve smoothing】, the dialog box shown in Figure 3.2, click OK. Before the change in the curve starts, left click, right-click when the curve ends the change, the dialog box shown in Figure 3.3, Click "No", i.e. in the figure shows the melting point of tin Teo. If the selected starting point or ending point is incorrect, you can click "Yes" to re-select when shows the dialog in Figure 3.3. Teo is the tangent at the starting point of maximum slope of the curve corresponding to the intersection of the tangent of the temperature that the melting point of the sample. Tm is the temp erasure corresponding to the peak, H for the sample actually measured enthalpy.

Figure 4.5 is DSC curves of pure tin, pure tin melting point is 231.9 ℃, instrument measures the tin melting point to be Teo = 231.4 ℃, peak temperature Tm = 250.7 ℃, the actual melting enthalpy H = 256.1952J / g.

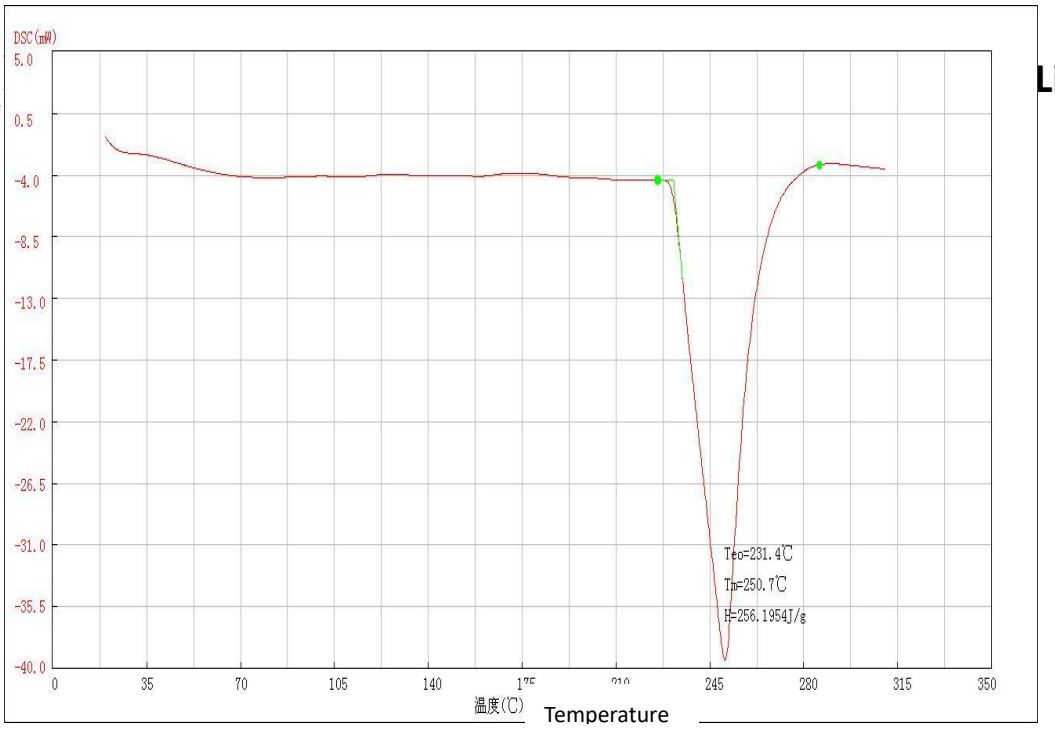


Figure 4.5

7.2 Coefficient of instrument

Because the instrument coefficient may change according to the environment, temperature, humidity, and so on will have a big or small impact. To ensure the accuracy of experimental results, we should always measuring instruments for the coefficients. Usually use tin, zinc, indium, etc. to calibrate instruments, measuring instruments factor.

Instrument calibration coefficients are the enthalpy of test calibration object under the premise of calibrated temperature, and then according to the theoretical enthalpy of calibration object and instrument coefficient calculation to calculate the coefficient of the instrument.

In the【Data analysis】column, select the【instrument Factor】, dialog box appears in Figure 4.6. Fill in the theoretical melting enthalpy and measured melting enthalpy the corresponding column. Click the calculation button to get the instrument coefficient. The instrument coefficient can be also used when calculating of the instrument the degree of crystallinity. If not continuous experiment, then you need to have the instrument coefficient recorded for later use.

Figure 4.6

In the pure tin sample experiments, for example, tin theoretical enthalpy input is 60.5J / g, measured enthalpy 256.1952J / g, the system calculates the instruments coefficient K that is 60.5/256.1952. The instrument coefficient software interface is generated automatically. As Figure 4.7

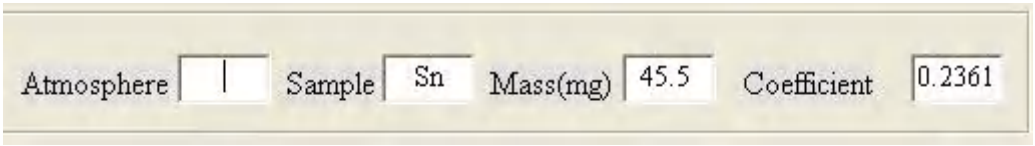


Figure 4.7

Determination of the coefficient is usually measured after calibration of instruments. At the instrument calibration, weighs the mass of the standard, fills in the quality column of real-time data column. If phase transition temperature after calibration is close to the actual temperature of the sample, then to record of the enthalpy, calculate instrument coefficients as the coefficients of the instrument.

7.3 Measurement of glass transition temperature

Glass is a substance into a glass-like amorphous body (glassy) process; glassy state is a state between liquid and solid. In this aspect there is no crystal structure. DSC determination of the glass transition temperature Tg is based on this nature that polymer at the glass transition temperature, its heat capacity will increase. In the DSC curve, and its performance: when through the glass transition temperature, baseline to the endothermic direction. Shown in Figure 5.1 Point A in the figure starts to deviate from the baseline points. Extend the baseline of the change before and after the change, the vertical distance between two lines is called gradient △J, In △ J / 2 can find at point C. From point C for the previous baseline tangent intersect at point B extension cord. ICTA proposed point B as the glass transition temperature Tg. Glass transition temperature, without a fixed value, with the measurement methods and conditions changes. Therefore, when marking a polymer' glass transition temperature, should indicate the measurement methods and conditions.

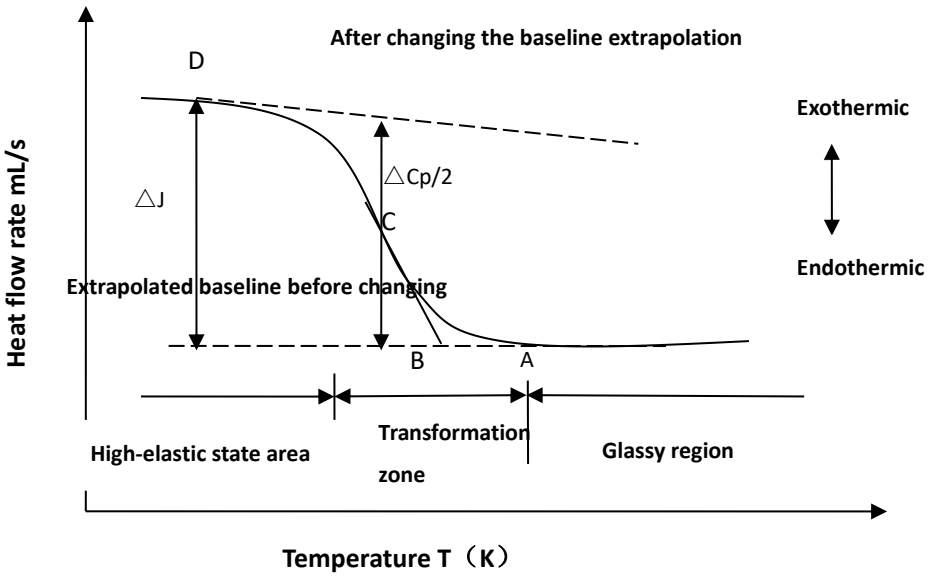


Figure 5.1

Procedures:

1. First, open the purge gas, the gas flow control about 120ml/min. Atmosphere is generally used as nitrogen, helium, argon and other inert gases,

some specimens can also be carried out in an air atmosphere.

2. Turn on your computer, the instrument data cable connected to the computer, plug in the power to the instrument, and turn the instrument on the back of the switch.

3. Click【Settings】in the menu bar, click【Communication Connection】, when showing the connection is successful, that means the instrument is connected to the computer.

4. Open the software, initial screen is oxidation induction period test interface; turn to X-temp interface for measurement of glass transition temperature.

5. In the【Settings】option, select 【Parameter Settings】, then dialog box appears as shown in Figure 3.1. The ending temperature parameters should be slightly higher than to the sample(needed to be measuerd) temperature. If the desired temperature range of the sample is unknown, the ending temperature can be set a little higher, generally 550 ℃, the heating rate is typically set to 10 ℃ / min, constant time 0min, can also be determined according to the specific operational requirements.

6. As needed to prepare a reference sample, an empty crucible generally used to make the reference.

7. Weighed amount of sample, usually 10 ~ 20mg, placed in a crucible.

8. Open the DSC furnace (guaranteed at room temperature); with tweezers put the aluminum crucible with tin particles in it into the center of the sample tray, and take another empty aluminum crucible as a reference. Close the lid.

9. In the experimental data field in the interface, input 【quality of the sample】, in milligrams. Click the  button on shortcut menu, the instrument starts the experiment.

10. This measure is the melting point of the sample, as a complete DSC curve peak appears, you can click the  button on the shortcut menu, stops the experiment. After the end of the experiment, on the shortcut menu click

button to save first the test data to avoid data loss.

11. After the end of the experiment, on the shortcut menu click button to save first the test data to avoid data loss.

12. While processing data, click on the shortcut menu button, open curve data need to be processed, click on the menu bar【Data Analysis】, select【curve smoothing】, the system can automatically correct curve.

13. Click on the menu bar【Data Analysis】, select【Glass transition temperature】, the dialog box shown in Figure 5.2, click OK. Before the change in the curve starts, left click, right-click when the curve ends the change, the dialog box shown in Figure 5.3, Click "No", i.e. in the figure shows the melting point of tin Teo. If the selected starting point or ending point is incorrect, you can click "Yes" to re-select when shows the dialog in Figure 5.4. After the end of the shift in the curve baseline, left click and right-click to take two points, dialog box as shown in Figure 5.3, Click the "No", If an error in taking the point, in dialog box in Figure 5.3, click "Yes" to re-take the point.

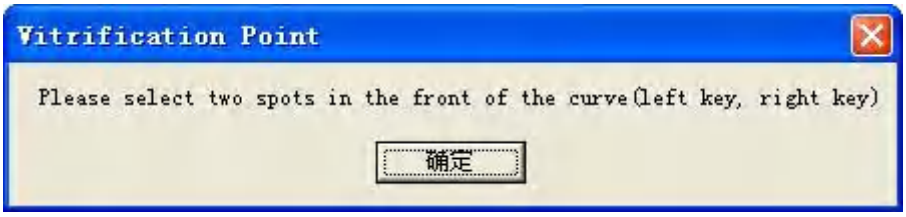


Figure 5.2

Figure 5.3

Figure 5.4

14. Figure 5.5 is the data obtained after treatment of a sample. The glass transition temperature of the sample Tg = 81.4 ℃, T1 is the initial temperature of the glass, T2 of the glass transition temperature of the termination.

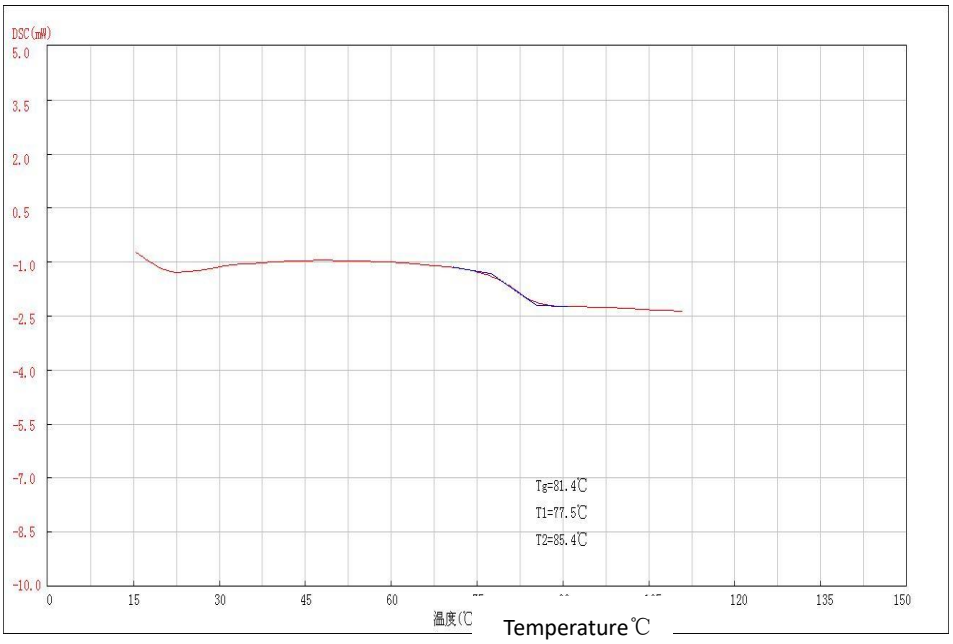


Figure 5.5

7.4 Oxidation induction period measurement

Oxidation induction time (OIT) is to determine the design at a high temperature (200 ℃) started to occur under conditions of oxygen autocatalytic oxidation reaction time, Is a measure of material in the molding processing,

storage, and use of welding heat degradation indicators. Oxidation induction time (referred OIT) method is a method using a differential thermal analysis (DTA) in the molecular chains of plastic heat reflection as the basis, to test plastic at high oxygen levels in the method of accelerated aging. The principle is that the plastic design with an inert reference material (such as aluminum oxide) is placed differential thermal analyzer, So that at a certain temperature rapidly replaced with oxygen chamber design an inert gas (such as nitrogen). Testing changes as the oxidation caused by the design DTA curve (DTA spectrum), and access oxidation induction (time) OIT (min), to assess the anti-heat aging properties of plastic.

Oxidation induction period method is a sensitive method for accelerated testing, Oxygen at high temperature polymer antioxidants completely consumed, rapid and highly exothermic oxidation reaction, With a differential scanning calorimeter (DSC) is an exothermic peak starting readily determined, so that a quantitative characterization of the time to understand the extent of oxidative degradation. Figure 7.1, A1, the instrument automatically switches gas, nitrogen gas is switched to oxygen, the oxygen starting point for the pass and start calculating the test sample oxidation induction time. DSC curve of constant temperature stage a line extending in the horizontal direction and the maximum slope of the rising edge of the exothermic peak at the point of intersection of the tangent point A2, A1A2 the oxidation induction period.

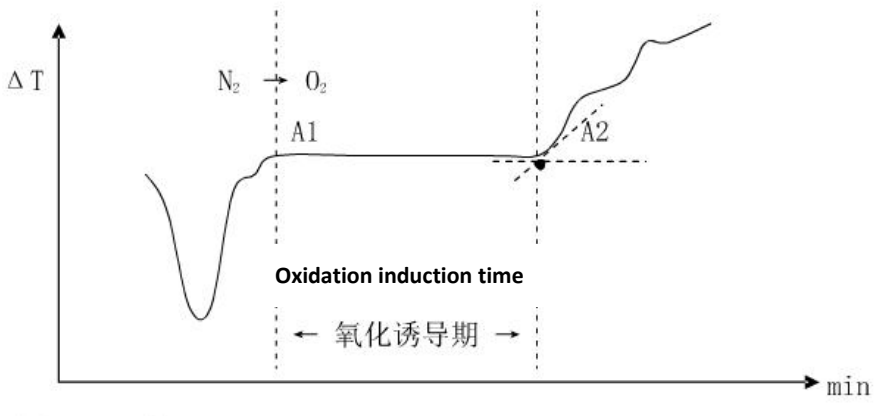


Figure 7.1

Procedures:

1. First, open the purge gas, the gas flow control about 120ml/min. Atmosphere is generally used as nitrogen, helium, argon and other inert gases, some specimens can also be carried out in an air atmosphere.

2. Turn on your computer, the instrument data cable connected to the computer, plug in the power to the instrument, and turn the instrument on the back of the switch.

3. Open the software interface for the initial oxidation induction test interface; click on the shortcut menu the  DSC-Temp key to the melting point (enthalpy) measurement interface.

4. Click 【Setup】 option in the menu bar and click 【communication connection】to show the connection is successful, the instrument connected to the computer.

5. In the【Setup] option, select Parameter Settings】, the dialog box shown in Figure 7.2. The termination temperature is 200 ℃, heating rate of 20 ℃ / min, holding time is 200min. Click "Settings", and then exit.

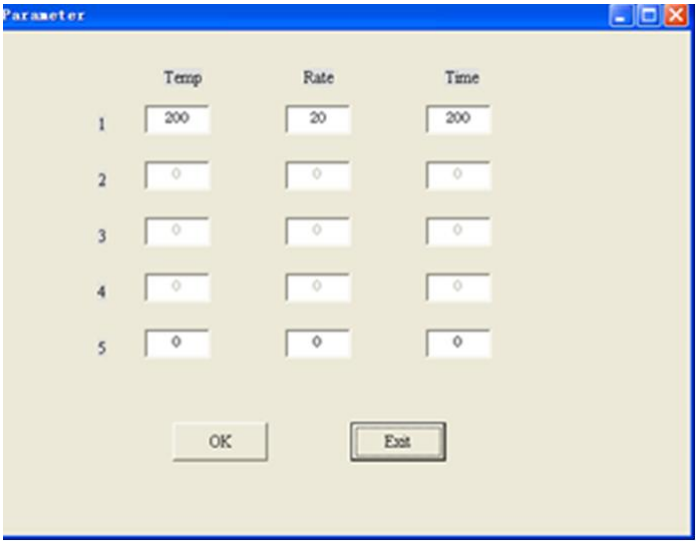


Figure 7.2

6. As needed to prepare a reference sample, an empty crucible generally used to make the reference. An inert substance can also be used.

7. Weighed amount of sample, usually 10 ~ 20mg, placed in a crucible.

8. Open the DSC furnace (guaranteed at room temperature); with tweezers put the aluminum crucible with tin particles in it into the center of the sample tray, and take another empty aluminum crucible as a reference. Close the lid.

9. In the experimental data field in the interface, input 【quality of the sample】, in milligrams. Click the button on shortcut menu, the instrument starts the experiment.

10. During the test, the instrument will automatically switch purge gas to oxygen, when a "pop" sound, slowly adjust the flow meter, the oxygen flow control in 120mL/min. After the DSC curves at the maximum heat release rate (ie, the inflection point of the DSC curve) and at least after another few minutes until showed significant exothermic effect, you can click the button on the shortcut menu, stop the experiment.

11. After the end of the experiment, on the shortcut menu click button to save first the test data to avoid data loss.

12. While processing data, click on the shortcut menu button, open

curve data need to be processed, click on the menu bar 【Oxidation induction period】, Dialog box shown in Figure 7.3 appears, click "OK", before curve changes, take at baseline two points, the dialog box shown in Figure 7.4, click "No", Dialog box shown in Figure 7.5, take two points at significant change place in the curve, Figure 7.4 shows the dialog box appears, click "No", If an error in taking the points, in Figure 7.4, select "Yes", to re-take the point.

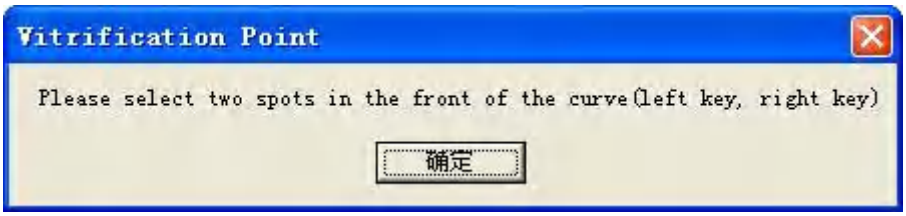


Figure 7.3

Figure 7.4

Figure 7.5

13. Figure 7.6 is experimental data of a sample oxidation induction, the upper curve of temperature profile, the bottom of the sample oxidation induction curve, the sample oxidation induction Oit= 66.97min.

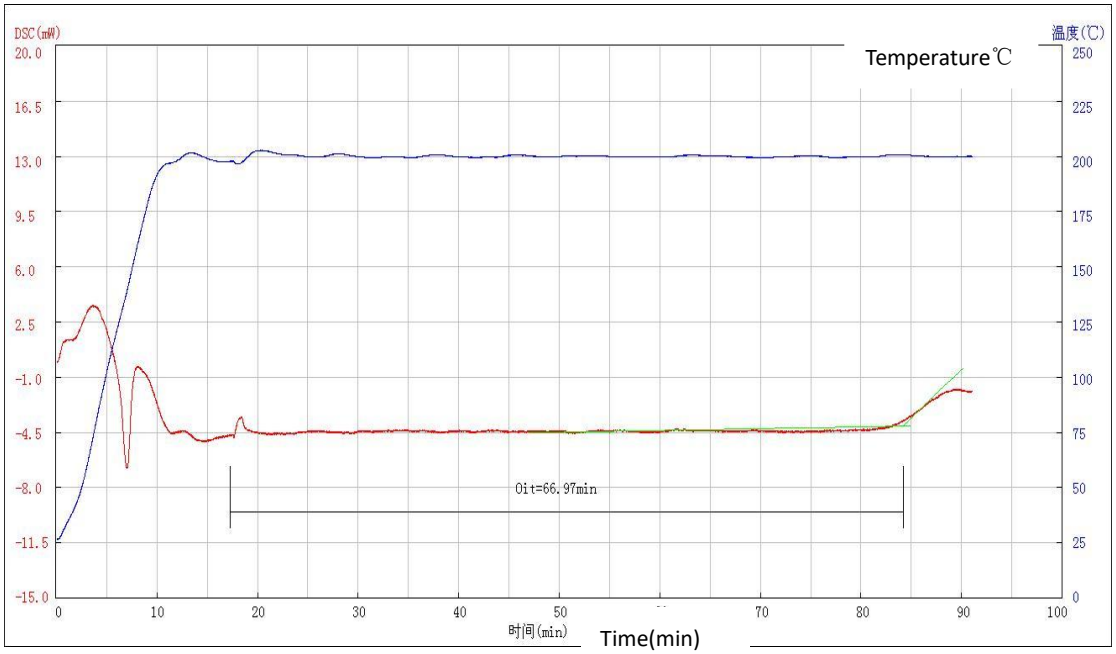


Figure 7.6

8. Instrument Precautions

1. In order to ensure normal use of the instrument, the sample can not occur within the temperature range from the thermal decomposition, do not react with the aluminum metal, no corrosion. If the tested sample is measured during heating to produce large volumes of gas, or to cause the instrument explosion, then can not be used. Therefore, before testing we should probably understand the nature of the sample.

2. Check if all connections of the instrument are correct, the adequacy of the gas used, the tool is complete.

3. In trial, if you choose aluminum crucible pans, the maximum testing temperature should not exceed 550 ℃. If in test the temperature exceeds the highest 550 ℃, you can use ceramic crucibles.

4. Laboratory temperature controlled at 20 ℃ -30 ℃, and the case of relatively constant temperature results higher accuracy and repeatability. The case of high temperature air conditioning needed to ensure the ambient temperature is relatively constant in the short term.

5. To ensure the accuracy of test results, before the use of the instrument first blankly burning (not putting any sample and reference material) for about 30 minutes.

6. Not using the instrument for a long time, when used again, be sure to do air burning two to three times, You can: Temperature set to 400 ℃, the rate is set to 10 ℃ / min, thermostat set to 0min, press the【Run】button.

7. Bottom of the crucible should be flat with no jagged or curved, or poor heat transfer

8. DSC sample preparation, do not sprinkle the sample in the crucible edge, so as not to contaminate the sensor, destroying equipment. Bottom of the crucible and the outer surface of all the samples can not be contaminated with impurities, to avoid affecting the results.

9. Quantity of the sample to be appropriate, not too much, not too little. Solid samples usually around 20mg. Liquid samples do not exceed two-thirds of the capacity of the crucible. If other requirements of the sample amount, upon request, to determine the amount.

10. The inorganic samples can be pre-grinding, sieving; For the polymer sample should be as far as possible uniform; 1 ~ 2mm fiber can be made of the same length; Powdered sample should be compacted

11. Crucible placed in a fixed position on the support vessel, quantity of the sample came to be spread evenly in bottom of the crucible, do not pile on one side; if the sample is granular, needs to be placed crucible central location.

12. Heating rate generally choose 10 ~ 20 ℃ / min. The curve drift if too large, lower resolution; too small then too long the time.

13. Do not use hard objects to clean the sample tray and experimental areas, so as to avoid causing irreversible damage to the instrument。

14. If the experimental area has powdery dust or other debris should use washing ear balls blown to clean, caution for mouth to blow and leads fans eyes.

15. If serious experimental area pollution , you can: Temperature set to 500 ℃, the rate is set to 20 ℃ / min, constant temperature to 30min, press the【Run】button.

16. In the process of data collection, should avoid the obvious shock around instruments, prohibited opening the cover, as slight touch in front of the instrument will produce significant peak on the DSC curve.

17. Do not adjust flow of purge gas in the process of collecting data, because the gas flow slight change may impact the DSC curve.

18. After the experiment, be careful of DSC lid, tweezers gently, avoid hot or lid damage.

19. Power supply: AC220V, 50HZ, power> 2000W.

20. Disconnect the cable; turn off the software before the instrument is closed to prevent online communication errors. (This issue in XP, SP3 system can be found and other systems have not be tested).

Solution: 1. If you encounter online success, no data is returned, you will need to restart your computer.

2. If you encounter connection fails, you will need to unload the USB device with an exclamation point in the Device Manager, reload it without the need to restart the computer.

Because of computers and equipment protection programs, sometimes prompt communication failure occurs when opening the software. When this occurs, turn off the instrument and the computer, and cut off the power, unplug that end of the instrument of data lines connecting computer to instrument, waiting for 3-5min, turn on the power, first turn on the computer, open the software, connect the data cable, open the instrument, wait about 20s, on the menu bar, click【communication connection】in the【Setup】options , after the connection is successful, press the 【Run】 button to operate the instrument.

Sometimes not once, may be repeated to operate the above operations several times. To avoid this situation, every time you boot, operate according to the above procedures (except the data line pulling our and inserting operation

9 Packing List

Host instrument	1
CD	1
Data Line	1
Power Line	1
Aluminum crucibles	100
Ceramic crucibles	100
Bag of pure tin grains	1
Fuse 10A	2
Operation manual	1
Warranty card	1
Certification	1

Remarks: Further counsel if other accessories are needed.

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