ai~Phase~
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Overview of commercial appaltus

Outstanding Features

Ai-Phase family is an overall measuring system for thermal properties of all materials from insulators to metals. Ai-phase mobile product line are high-performance machines full of originality, designed under 9 concepts as follows:

1. Wide temperature wave frequency — from 0.001Hz to 2 kHz
2. High Speed Response Sensor and Heater — in-house development
3. Thin and/or Small sample — μm, mg order
4. Short measuring time — several minutes order
5. Compact size and light weight — easy hand caring and transportation (delivery service shipping and repair)
6. Easy maintenance — units are easily changed for new one
7. Without experience — full automatic, user friendly software
8. Saving energy design — USB bus power - drive
9. ISO standardized method — ISO22007-3 and -6

1 :   Let’s start

Please connect

Main Amplifier,
Sample Holder,
Power supply by USB
and PC

then
Switch on

heater

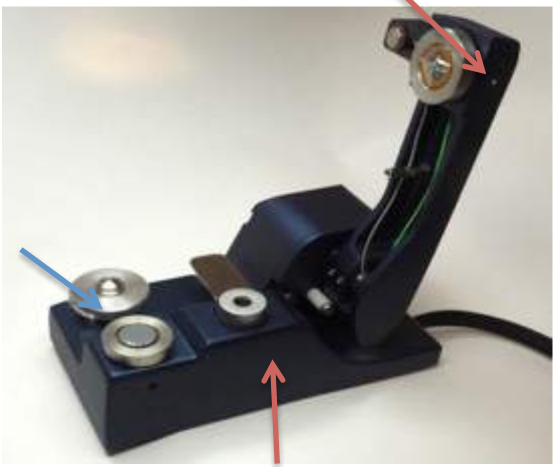
Precautions for handling of Type 1

- In principle, Type1 machine has specifications suitable for measurement at room temperature.
- Power is supplied when connected via USB, so no power connection is required.

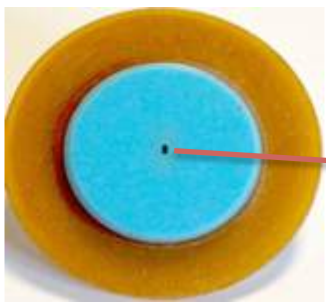
- The orange protective ring prevents the sensor from being damaged due to the arm falling.   Remove it.

- Lift the arm by hand and slowly move it to the other side. Slowly when lowering, too.

Arm stopper



Thickness meter



sensor

The black dot in the center is the sensor.
0.5x0.2mm

Set up of device

- M3 type1 is driven by a USB bus power.
- However, if you do not use temperature controller, it will work only with USB bus power.

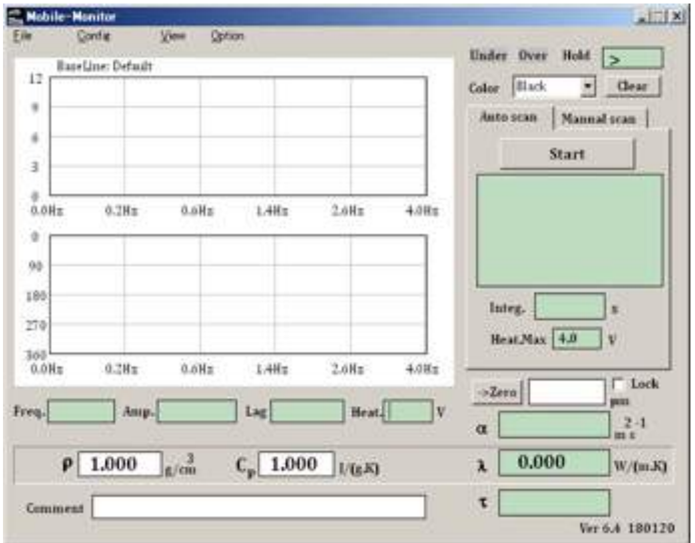
- The arm stopper may be used at the shipment to avoid trouble during transportation.

- (Note) If the red lamp is lit, it means that the arm has stopped at a certain distance.

- Turn the dial as clockwise and lower the arm until the LED lamp goes out.

- The liquid crystal display on the main unit is as shown on the left.

Launching the software



- (2) Connect the main unit to a PC with a USB cable.
- (3) Switch on the power of the device.
- (4) Open the EXE file in the main folder. The measurement screen(auto mode) appears.
- (5) The LCD screen of the main unit changes to “tc_get”.

  (6) Hold for 10 minutes or more for warming up.

The driver is on the DVD. Please install if necessary.

Reset of this device with the power button.

USB port recognition differs depending on the PC model and Windows type.
Details are on the next page.

Baseline check before start
(manual mode)

Defined from4 to 400Hz

- Check the temperature setting. Press the “manual mode” .
- Put a weight on the tip of the arm.
- Measure under conditions of described in this figure.

Press the “start” button.

- The upper plots is the amplitude(arbitrary unit, log scale) and the lower is the phase(deg.) described as a function of frequency.

- At this time, make sure that it matches the baseline (solid line). If even one point does not match, cleanup the surface of the electrodes and try again.
- If they still do not match, refer to chapter 5 and 4.

- This procedure also serves as a warm-up, be sure to execute it when starting up.

- You can stop in the intermediate of measurement.

Initial Parameters of automatic measurement for this device

Open the option column and choice "StratUp"

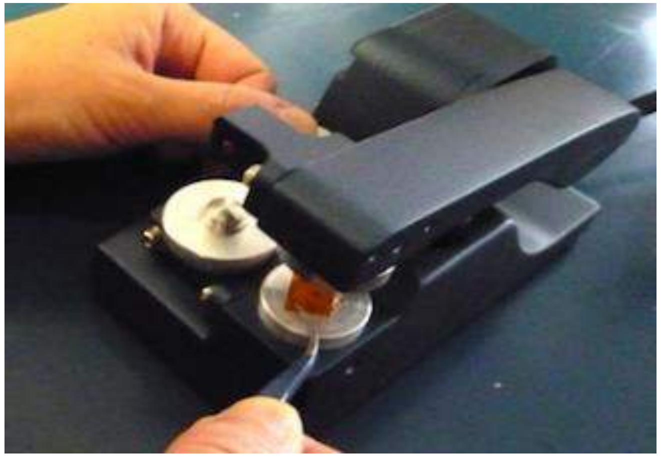
This is the initial set of parameters of auto measurement.

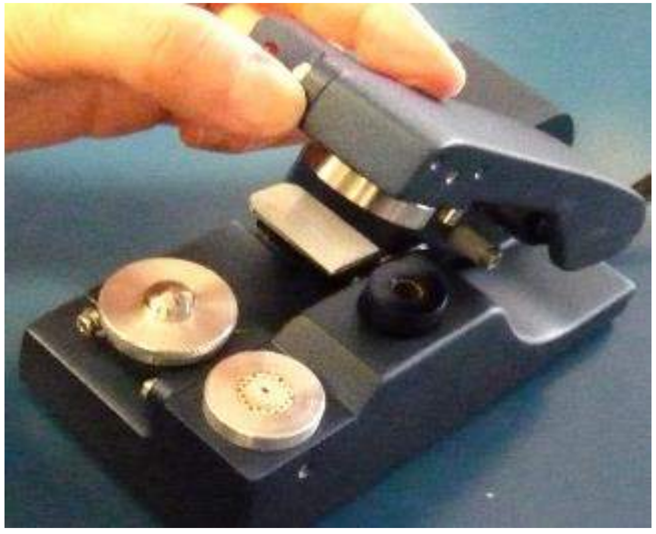
PhaseLag1, 2 are important for automatic measurements.
Lag1=170-200, Lag2=210-240

Fine adjustment becomes necessary when heat diffuses in the plane direction, such as the oriented or heat conductive sample.

If the start frequency is changed depending on the sample, the measurement time may be shortened.

Device check (auto mode)

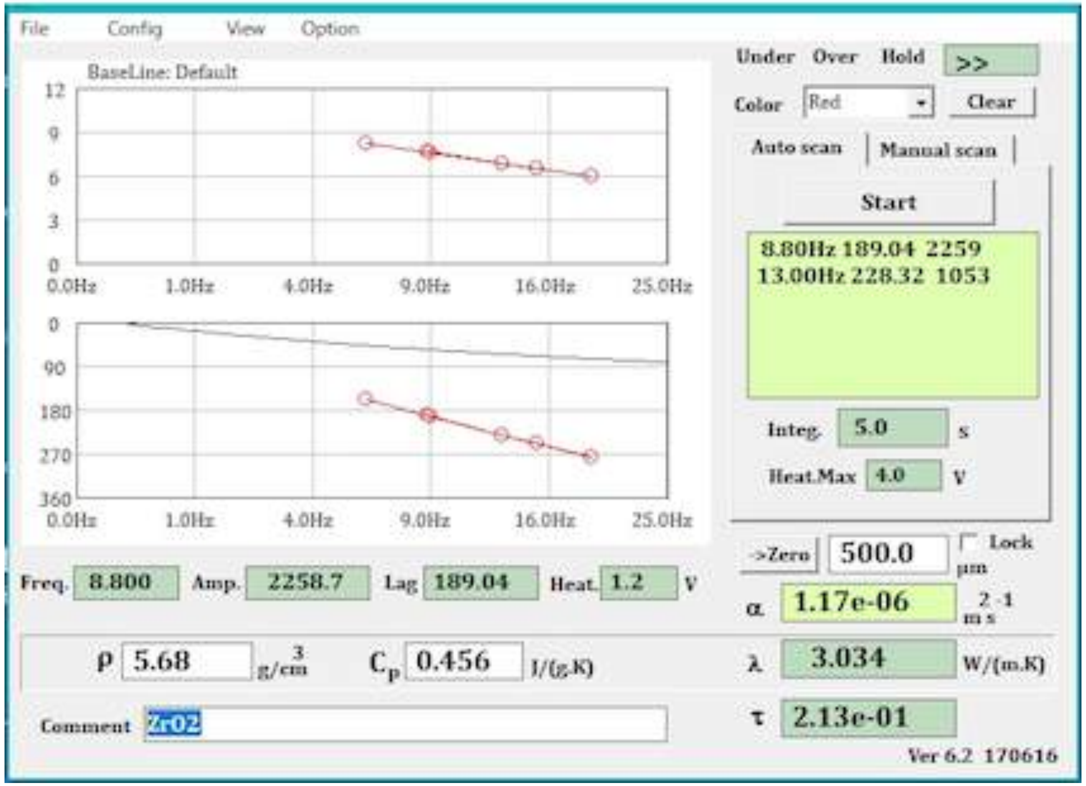




- (1) Zero reset have to perform without sample.
- (2) Place a weight on the tip of the arm to close contact.
  100g for normal, 50g or not use for soft material.
- Be sure to remove the weight when raising the arm.
- (3) Push down the left pedal and the arm will rise slowly. Insert the sample and release your finger. Or lift the arm by hand and slowly move it to the other side.
- (4)Set the sample in the center black part.
- (5) Put the weight on again.
- (6) Wait for about 10 seconds until it stabilizes.
- (7) Press the auto mode and press the start button.
- (8) You will be notified by sound when the measurement is complete.

- The standard sample of this device is attached
- PI (Kapton500H) film of 0.125 mm.
- Diffusivity is ca.1.18x10^{-7} (room temperature) ,
-

Test data for zirconia 500 plate

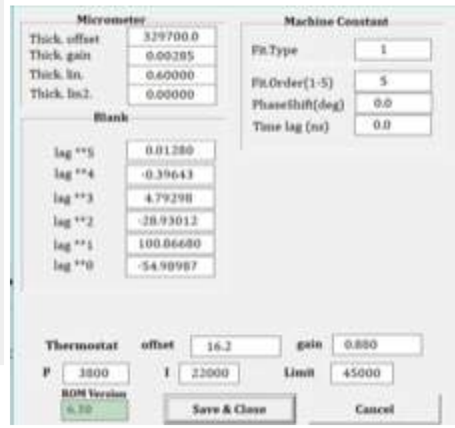


First, measure the attached zirconia plate in auto mode. The thickness is exactly 500 microns. The slight deviation is due to the deformation of the PET protective film, etc. Also, since it is a differential transformer system, it may change over time due to changes in the environmental temperature. It fluctuates between 495-505 as a 1% error.
The thickness can also be entered manually. You can lock it, for example by using a micrometer value.

Make sure that the plot is linear on the screen during zirconia measurement and that the calculated thermal diffusivity is around 1.15 to 1.24x10-6m2S-1. The solid black line is the baseline value of the blank, which is the phase lag (device constant) derived from the sensor, heater, interface resistance, protective film, etc.
After turning on the power, it will automatically become the default baseline.

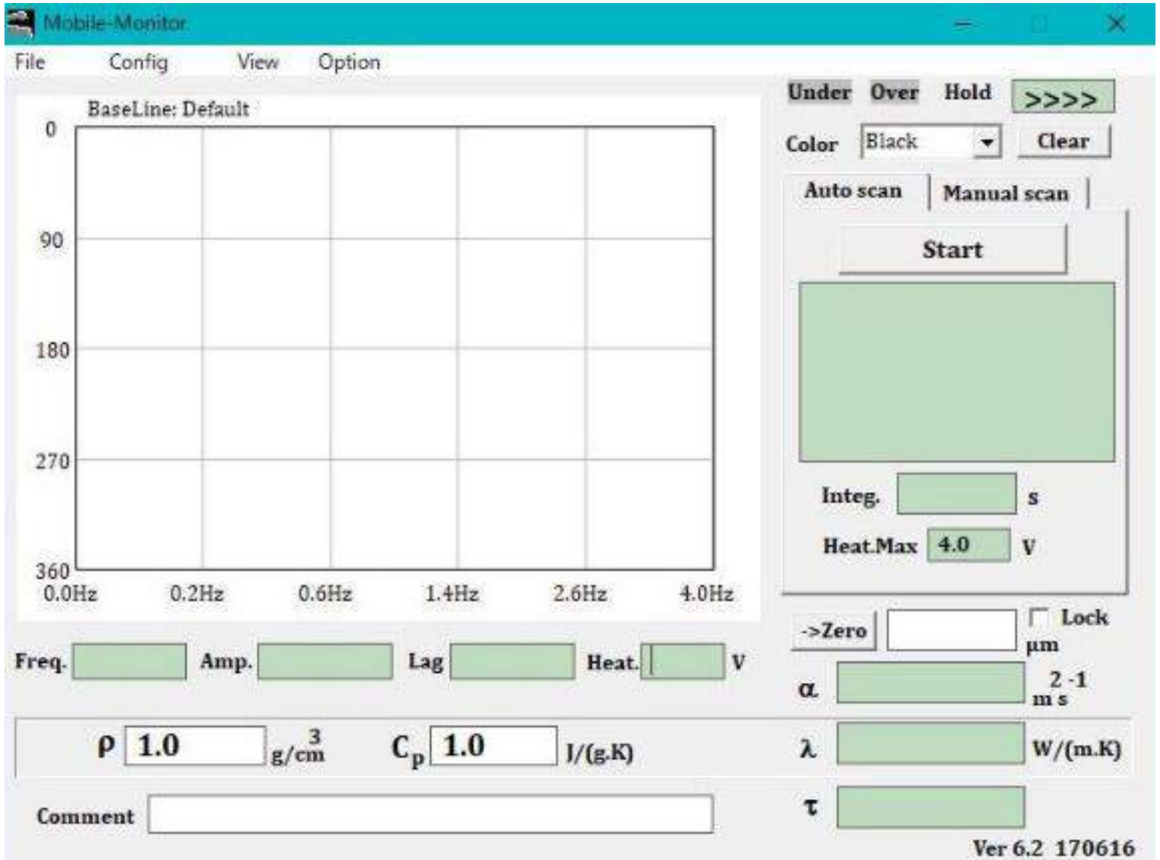
The frequency, actual frequency, and amplitude of Lag1 and Lag2 are shown respectively.
The sensor sensitivity depends on the temperature, but the amplitude is proper around 1000.

2. Screen of software



You can also use a sound to indicate during the measurement. ai-phase sound signals the progress and completion of the plot. This is the work of Kunihiro Kawano, a composer who is also a founding member of our company.

Software screen　Auto mode



Measurement status
Color selection of plots
Choice of automatic or manual
Push for start

Data table

Integral time for measurement
Limit of applied voltage
Zero reset　thickness*　lock(check)
Measured diffusivity
Calculated conductivity　α・Cp
Relaxation time from the gradient
= measured value

Upper　Measurement status
Middle　Density*　Specific heat* from keyboard
Lower　You can write　comments at any time

$\alpha = (\text{thickness})^2 / \tau$

* you can write arbitrary value

Auto measurement parameters

USB port

IntegTime normally 5sec 50sec used low frequency.

Start Freq. Free. Depends on the baseline type.

AmpMin The voltage automatically increased up to this limit.

PhaseLag1 Gives low frequency phase. Ex 190 (170-200)

PhaseLag2 Gives high frequency phase. Ex 230 (210-240)

Retry normally 10

FreqStep automatically increased if necessary

StartVolt normally, 1V, automatically increase if necessary

LimitVolt limit voltage in automatically increase

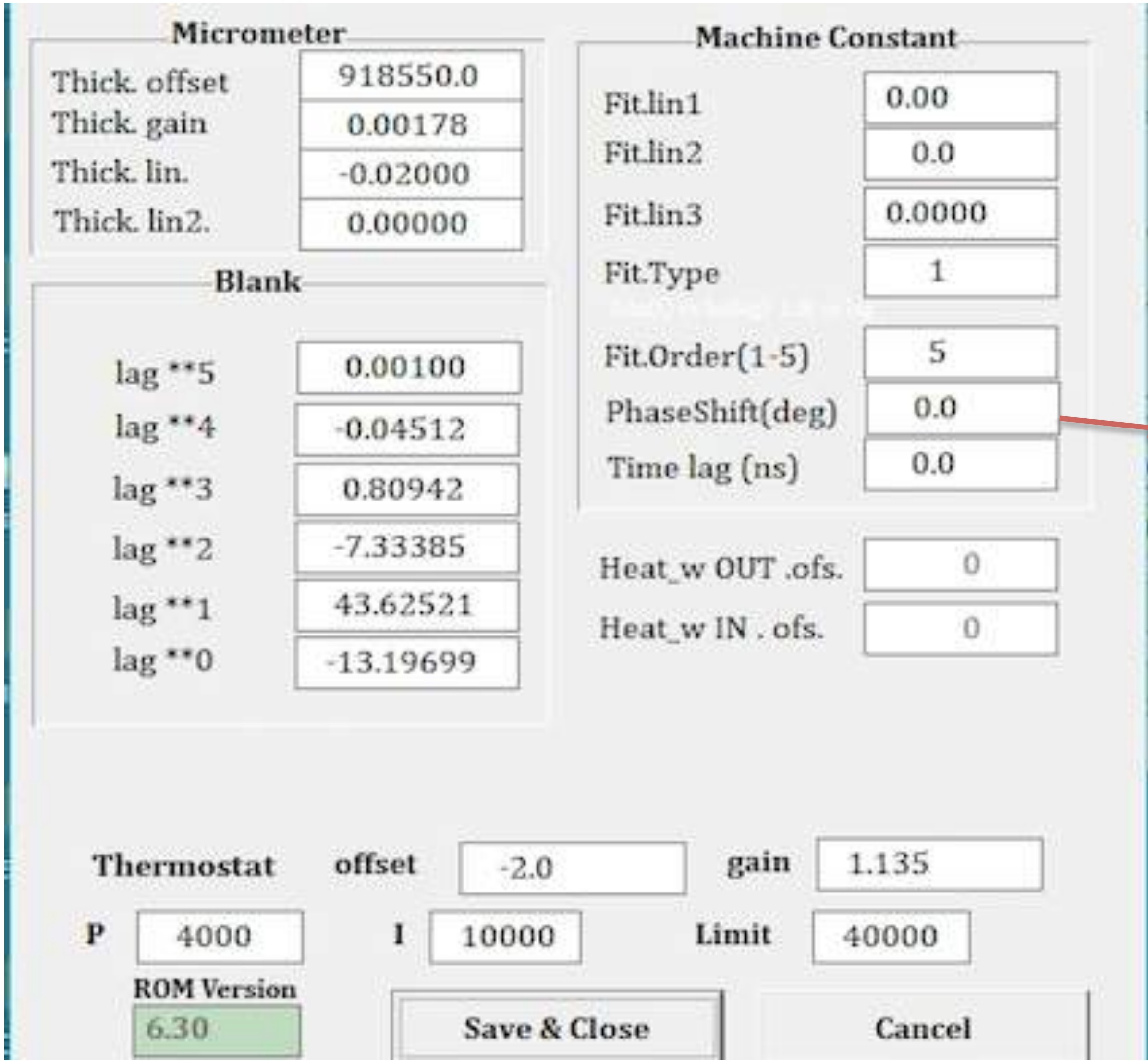
TWG **Check** to keep the micro heater on all the time.

Logging FileName the file name of the auto log.

Configration

Thickness gauge

Baseline
fitting parameters

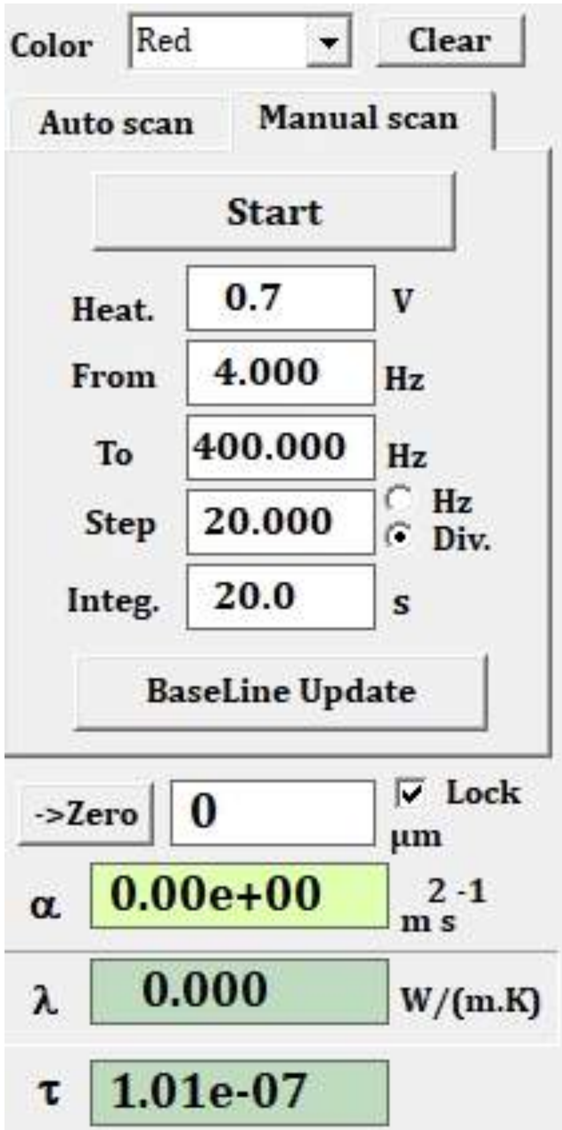


Baseline function

Usually 0,
60 for thick samples

Not used in M3 type1

Parameters in Manual Measurement



- Manual measurements are essential for baseline measurements. In addition, the number of integrations is larger and the accuracy is improved compared to automatic measurement.

- The initial settings of this device are as shown on the left. Two frequencies specified by Auto are entered, and it can be freely determined based on 1. 2V, 20 divisions and 20 seconds of "Integ." time.

Preparation of reference materials

- There is no authorize standard material for thermal diffusivity. We use zirconia 500 microns (manufactured by Mitsutoyo), which has a stable thickness as standard.

  The thermal diffusivity is about $1.18 \times 10^{-6} m^2 s^{-1}$ around room temperature.

- We recommend that you decide on your own standard sample and judge by comparing with these standards.

Examples(room temperature)

Glass	～4.5～$x10^{-7}$ m^2 s^{-1}
Polyimide Kapton	1.0-1.2$x10^{-7} m^2$ s^{-1}

For thickness calibration

Steel thickness gauge 0.1mm, 1mm etc.

PI film of 7.5, 25, 50, 75,125 micron meter.

3: Thickness Gauge

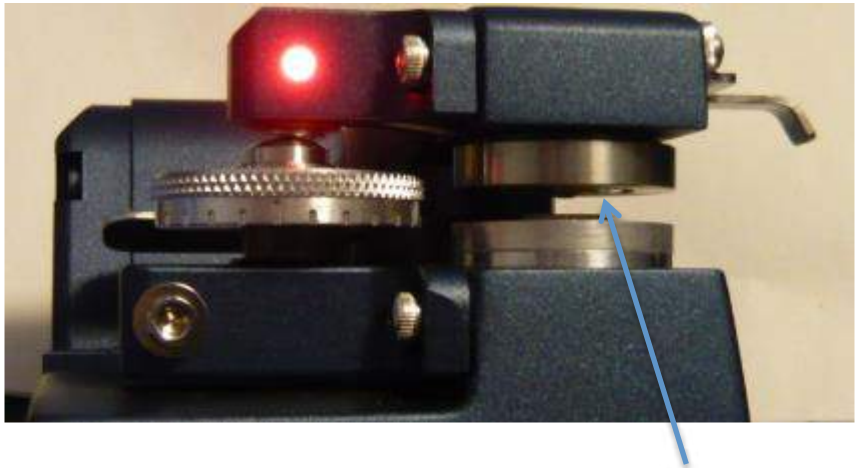
First, press the zero point.

Second, insert the standard sample and press the auto button.

Check the thickness.

If there is a difference of 1% or more, adjust according to the following procedure.

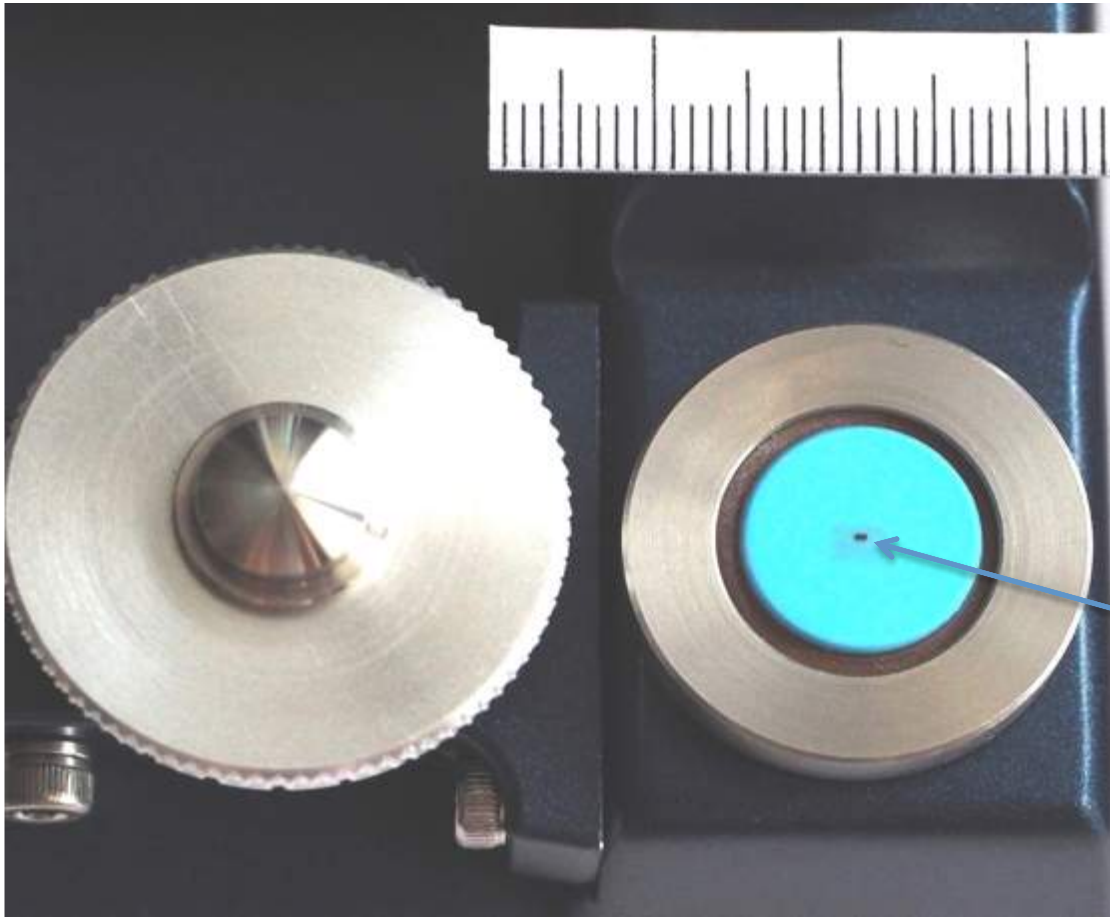
Arm stopper for keeping constant distance



Red LED lights up
during operation

- The arm stopper acts as a spacer that provides a gap between the micro heater and the sensor.
- This is applied to the measurement of liquid, powder and molten samples.
- Turning the dial clockwise lowers the stopper (reduces the gap) and raises it counterclockwise (larger gap).
- It is about 0.5 mm with one rotation of the dial.
- When the arm and stopper come into contact, the lamp lights up and the gap is fixed. The height is measured with the thickness gauge of the main body
- **At the end of measurement, raise the arm, turn on the lamp, and then turn off the power. You can prevent damage due to collision during transportation.**

4: Sampling and Measurement



Sensor size
0.5x0.2mm

Precautions for data acquisition

- 30 minutes after turning on the power. Temperature field instability due to initial heat generation
- Confirmation of baseline Measurement and replacement under the conditions of use.
- Adjust the film thickness gauge firmly, because there is a drift.

- Carefully pinch dust, surface scratches, sample warp
- 10 seconds after sandwiching the sample and probe to avoid a creep phenomenon.
- Thermal diffusivity measurement uses the thickness measured last
- Measured 3 times without changing the placement conditions at all. Confirmation of stability
- Measurements least 10 times in one sample, and the prospect of 30 times for precise measurement.
- Exclude data that deviates significantly from the average and do not add it to the average calculation.

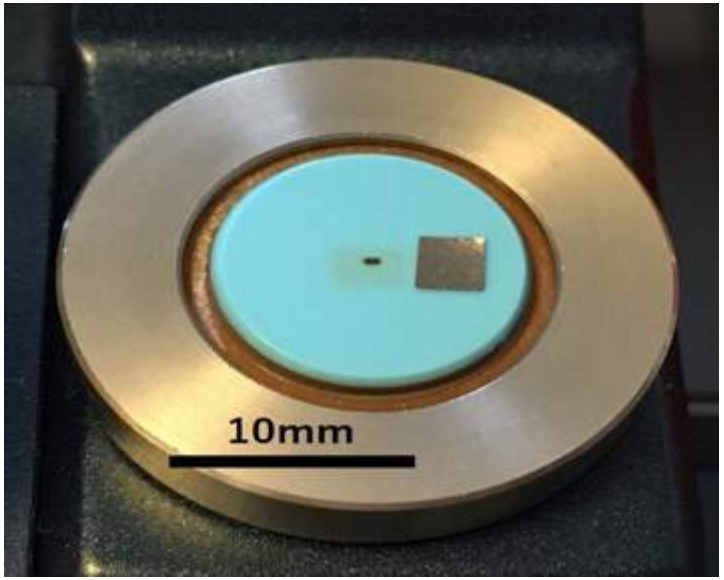
Sample setting

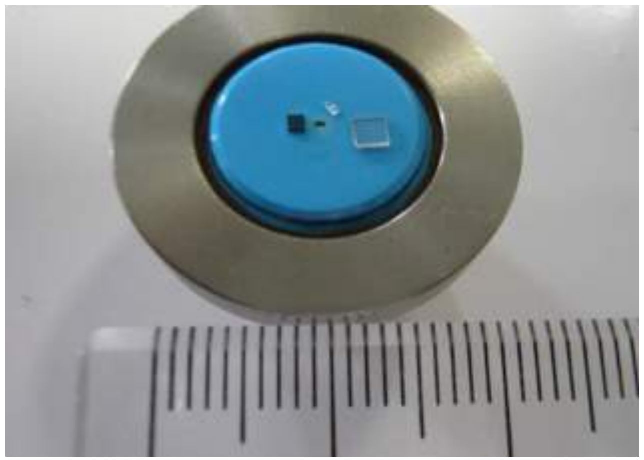
- Cleaning the sample surface
  And apply a very thin layer of grease on both sides.

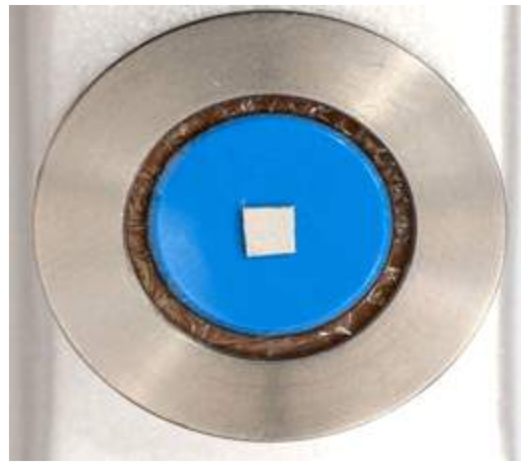
- Lower the arm without sample and take the zero point.

- Place the sample so that it contacts the sensor (black dot)
  Put the weight on the tip of the arm.

- Set to auto mode and press the start button.

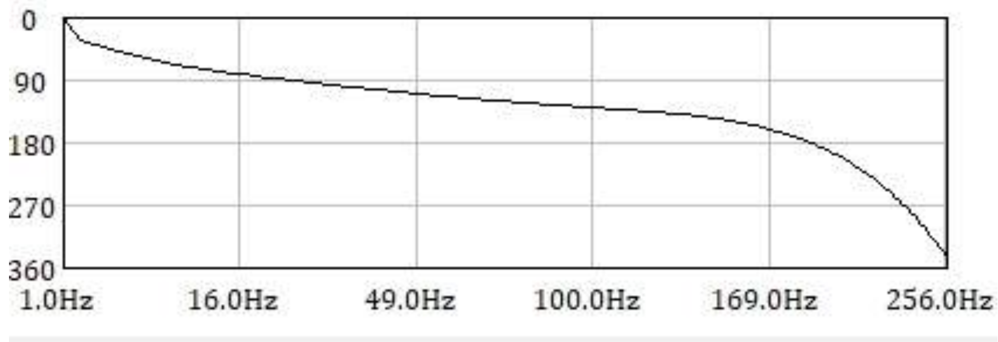






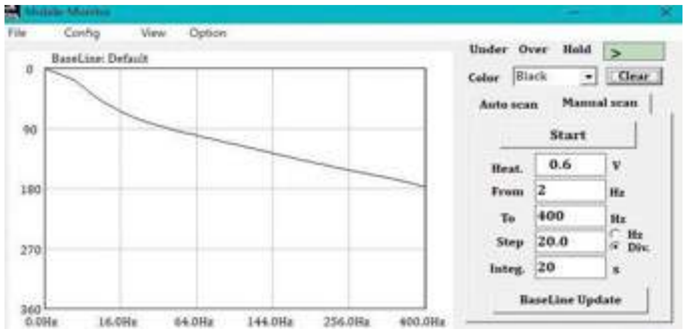
5. Base Line

- The baseline affects the phase by sensor, heater, wiring materials, protective films, interfaces, etc.
- The baseline is shown as an approximate curve (solid line) by a polynomial function. In practice, there is a valid domain.



- The actual measurement data is calculated by subtracting the baseline.

- Baseline is very important. Do not hesitate to retake the baseline at any time.



Baseline determination

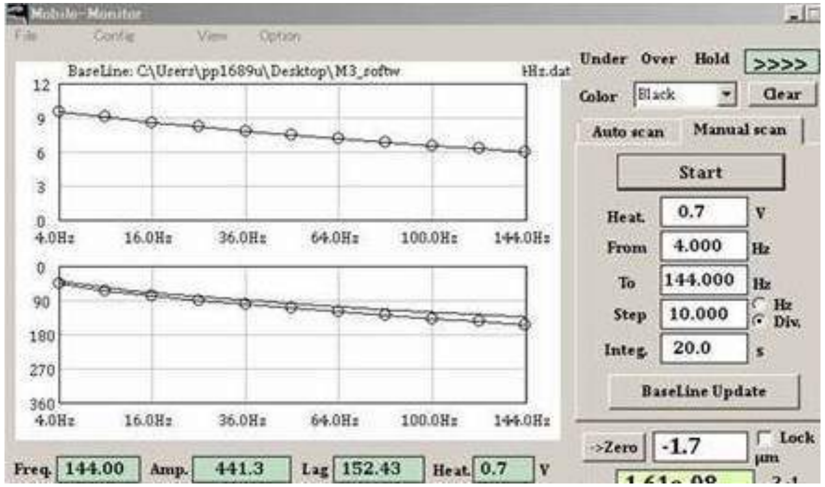
- You can change the baseline at any time as it will change depending on the weight.
- The solid line is the baseline (default) stored in the ROM in the device, and is automatically called at startup.
- Be sure to perform baseline measurements manually and at a low voltage without putting a sample.
- For general purpose, take a wider frequency band, and for strict measurement, take the same frequency as the actual measurement every time.

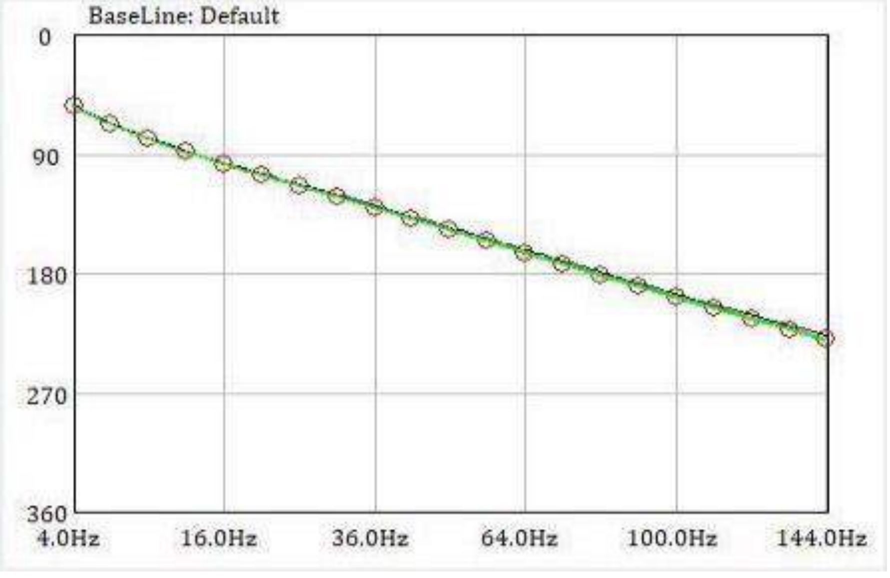
- At least 8 measurement points are required.

- The measurement conditions depend on the sample. The following are three typical conditions.

- Baseline determines the success or failure of the measurement

Base line check





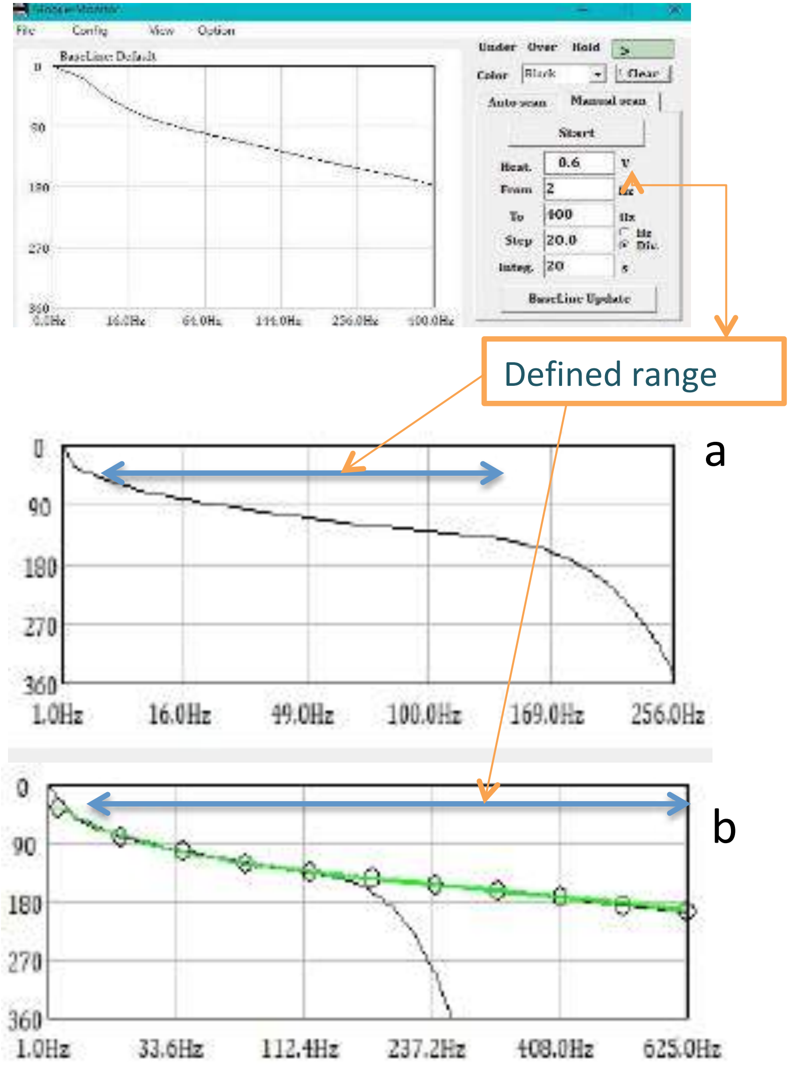
- At this time, make sure that it matches the baseline (solid line).
- (Upper) If the blank measurement does not match the solid line , it means that the surface is dusty or dirty. Please clean it. There is also a change over time.

- It will be possible to display only the phase in View.

(Under) There is a slight discrepancy in high frequency range. You need to retake the new baseline.

- We recommend that you change it as shown in the figure on the left.

- If in doubt, when the value of the standard sample is not stable, re-baseline without hesitation.



Baseline determination

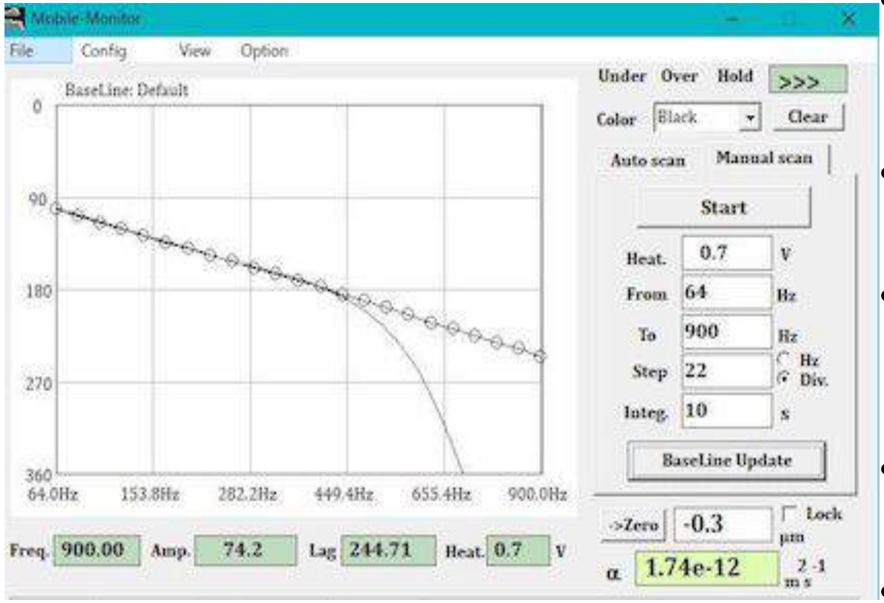
- The solid line is the baseline (default) stored in the ROM in the device, and is automatically called at startup.
- The acquired baseline can be named and saved.
- Be sure to perform baseline measurements manually and at a low voltage without a sample.

- For general purpose, take a wider frequency band.
- “a” will not be a straight line high freq. “b” is an expanded
- domain. Measurements should always be made within the defined baseline range.

- For accurate measurement, take the same frequency range as the actual measurement. It is acquired under the same conditions except for the voltage.

- At least 8 measurement points are required.

- **Baseline determines the success or failure of the measurement. Please take care!**



High frequency (thermally thin sample*)

- If you want to get a high frequency baseline, increase the start frequency. This makes the approximation linear.

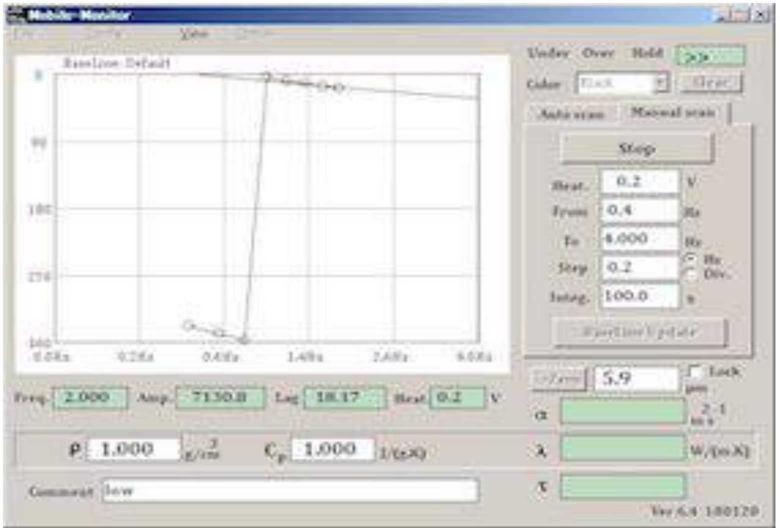
- Set the heat voltage slightly higher than the standard.

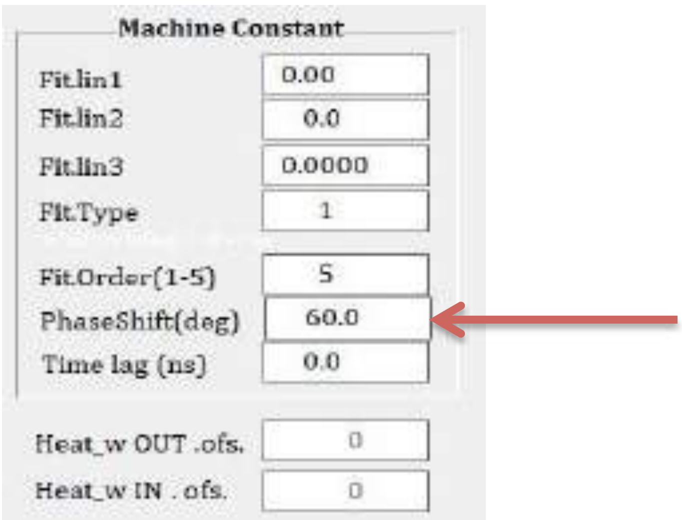
- The approximate straight line extends to the high frequency side. The specified range is from 64 to 900Hz

- Without a protective film, it can measure up to 1200Hz.

- Don't forget to save it with given name.
- Of course, you can save it as the default.

* Thermally thin means a sample that is actually thin or has a large thermal diffusivity at the same time. It refers to the state in which heat is immediately transferred to the rear surface.



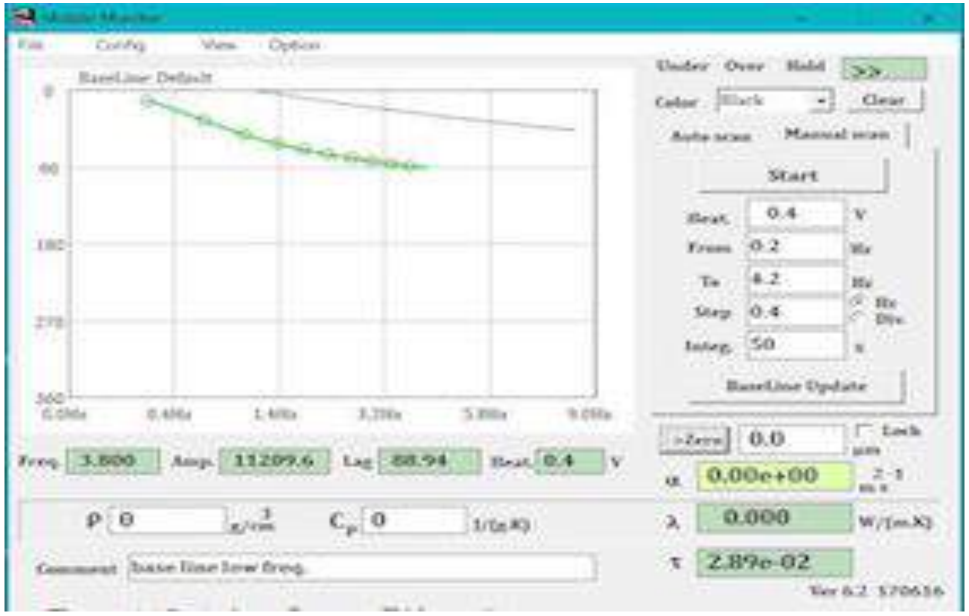


Low frequency (thermally thick sample)

- If you get the baseline at low frequencies under normal conditions, the plot will skip as shown.
- If there is such a jump, it becomes impossible to calculate.

- Shift the entire plot downwards to secure a straight line.

- On the config screen, enter 30-60 ° in “ shift” to lower the first plot as don't over scale.



Low frequency baseline conditions

- The voltage is lower than standard. 0.3V
- The frequency is about 0.2- 4.2Hz, but the range is arbitrary.
- The time constant should be 50 seconds or more.

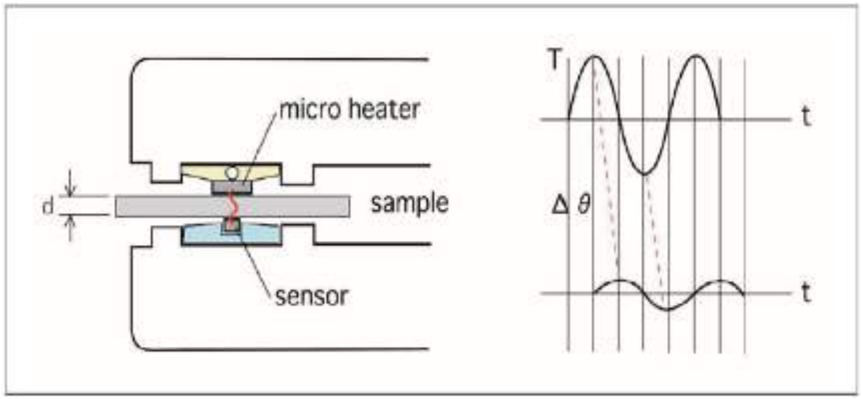
- Normally, div is used, but “<u>deg”</u> is also effective at low frequencies.
- In this case, 4Hz is divided by 0.2Hz, so 20 points are obtained.
- Don‘t forget to put “deg” back when returning to normal measurement.

- For the measurement, add 60 ° to each of the two phases of the setup.
- Don't forget to restore the original value.

How to determine the frequency

$$\Delta\theta = -\sqrt{\frac{\pi f}{\alpha}}d - \frac{\pi}{4}$$

$\Delta\theta$ [rad]: phase delay
f [Hz]: frequency of temperature wave
$\alpha[m^2s^{-1}]$: thermal diffusivity
d [m] : thickness of the specimen



- Theoretically, stable measurement is possible at high frequencies above a certain level. However, at high frequencies, the signal becomes weaker and the signal-to-noise ratio decreases significantly.

- The appropriate frequency is kd around 3. If the thermal diffusivity and thickness of the sample are known, the frequency can be estimated using their relationship.

- kd= $\sqrt{\frac{\pi f}{\alpha}} \cdot d$

- We have—independent of the surrounding conditions—the following relation: kd is above 3-4.
- Here $\Delta\theta$ is the phase shift, α is the thermal diffusivity, *f* is the frequency, and *d* is the sample thickness. Thus, plotting the phase shift versus the square root of the frequency, and using separately measured values for the sample thickness, allows calculation of the thermal diffusivity.

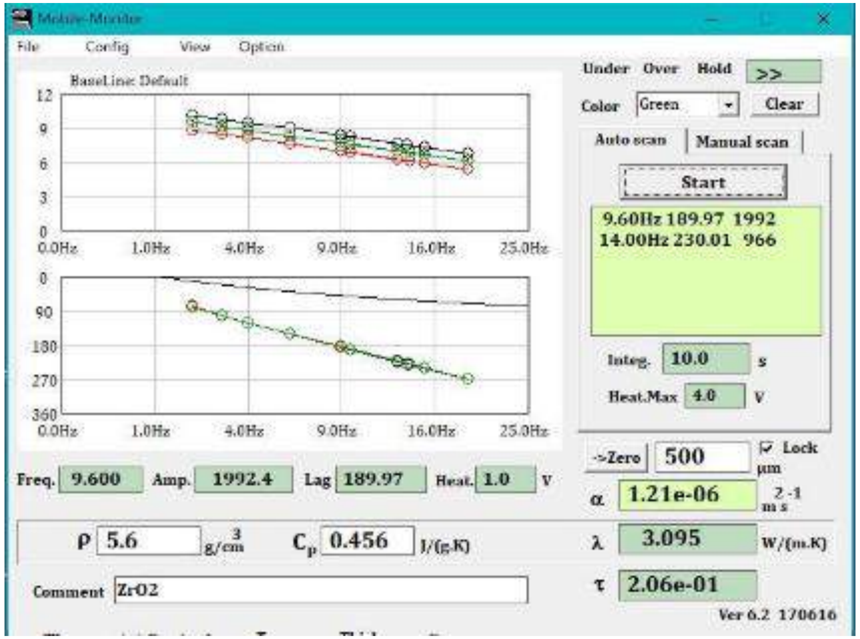
6: Maintenance

- TWA is a simple method, but it is essentially a precision measurement. The best maintenance is indispensable.

- Let's check the condition before measurement !

- Dirt is a great enemy of this measurement !!

- The sensor is a brittle ceramic, so please treat it carefully !!!



Data check by standard material

- Be sure to check when starting the measurement.
- It can be seen that even if the voltage is different, the amplitude is reflected but the phase is not affected.
- It has been confirmed that the attached standard sample at the time of shipment is
- $1.20 \pm 0.05 \times 10^{-6}$ m^2s^{-1} at room temperature using 0.5 mm of zirconia plate. It depends on the purpose and device.

- The main reasons for the disagreement are
- (1) Noise----measure again
- (2) Effect of dirt and dust----cleaning
- (3) Poor contact----sample problem, position change
- (4) Baseline change---- reacquisition
- (5) Protective film trouble-----Exchange
- (6) Machine trouble----consult with ai-Phase Co.

- The recommended standard substances; glass plate, pure metal, polyimide film, and your special materials.

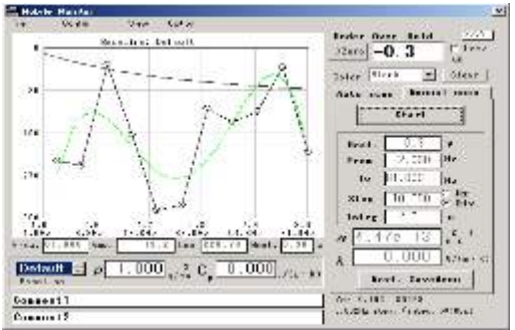
Daily care

- Make sure there are no residual particles or dust.
- To clean the electrodes,.first, it is recommended to wipe the surface with your soft fingertips.
- You can also wipe it with a soft cloth. For heavy residues, it is also effective to add water or ethanol etc. PET film is used as the protective film.

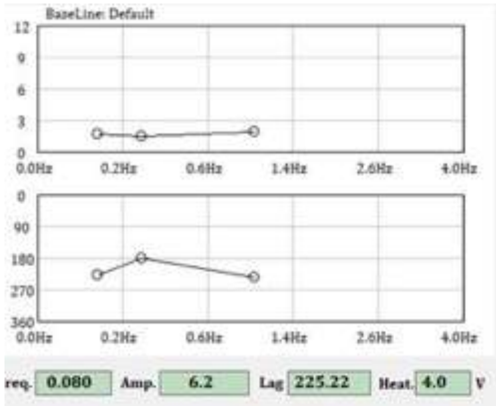
- Tissue paper is not allowed, because cellulose is a hard material.

- Before the test start,
- Checked the surface by stereomicroscope or loupe.
- Check the thickness gauge.
- Check the baseline.
- And check the equipment for measuring standard substances.

Failure diagnosis and check



- The most common failure is electrode damage. Depending on the degree of failure, the amplifier becomes smaller and it becomes unstable. The phase plots are also not linear.



- The plot will not be stable as shown in the figures.
- Replace with a new one.

- Other factors include heater disconnection, thickness gauge failure and board failure. Power supply failure can be dealt with by adjusting a DC12V2A converter.

- The sensor can be removed by turning the cap. It is convenient for cleaning and attaching a protecting film.
- There is a distinction between top and bottom.
  Please be careful when returning.

- The electrical resistance of heater ca. 100 ohm
- The electrical resistance of sensor ca. 2-3 kohm