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Detailed Technical Specifications for Thermal Conductivity Measurement Instrument By Flash Method

Hardware

Design, Furnaces, Sample Temperature, Atmospheres

- The instrument shall be Gas tight and designed as table top instrument for flexible application. Sample handling should be performed within this footprint. Beside thermostat and optional LN2 vessel all parts should be integrated in the device. No additional boxes for electronics or power supply etc. should be required.
- The instrument should have vertical set up with a light flash arranged on the bottom, the sample in the centre and the detector on top. No wave guide should be required guiding the flash from the reflector to the sample.
- The distance between sample, detector and flash lamp should be minimized to get an optimum signal-to-noise ratio. No specific sample preparation (e.g., silver paint) should be required if the sample material is not electrically conductive.
- The instrument should allow measurements from -100°C … 500°C within one instrument and without any change of detector, furnace or other components
- The thermal mass and total volume of furnace must be minimized to get a fast temperature stabilizing and short measurement times.
- Heating rates up to 50 K/min should be possible.
- The temperature equilibrium should be determined not only from the sample temperature signal but also from the stability of the detector signal.
- The furnace should be capable of holding the temperature stable to at least ±0.1 K at a given pre-set temperature after achieving each isothermal temperature segment (during measurement).
- The temperature accuracy of the furnace should be ± 0.1 K.
- The isothermal stability should not exceed 0.02 K/min
- The sample temperature will be measured using a type E.
- For furnace cooling, a cooling system (LN_2) should be available. The LN_2 controller is optimized for fast temperature stabilization with a reduced LN_2 consumption.
- The furnace shall be capable of running samples under oxidizing, reducing and inert atmospheres.
- Warranty for complete equipment for a period of 12 months from date of installation.

Detector, Thermal Diffusivity Range, Accuracy

- The system shall have the capability of being equipped with liquid nitrogen cooled MCT detectors with a nominal 24h-Dewar. IR detectors are capable to measure samples which are highly conductive, inhomogeneous or enclosed in a container allowing non-contact measurements.
- The exchange of the detector should be simple and executed by the operator within a couple of minutes.
- No iris or any additional orifices shall be used, for reducing the energy towards the detector.
- No mirrors shall be used on the detector side.
- The instrument should have the capability to measure thermal diffusivities over the range of 0.01 mm^2/s to 2000 mm^2/s between -100°C to 500°C. For most materials it should be possible to achieve accuracy better than 3% for samples with optimum thickness and diameter.
- The instrument should have the capability to measure the specific heat of solids with an accuracy of better than 5% (for standard materials).
- The detectors of the system shall be user-exchangeable to accommodate any future application.
- An automatic LN_2 refill system for the detector must be available as an option. The nominal operation time of such a refill device should last for more than one (1) week.
- No compressed air shall be required for actuator control (no pneumatic control) for filters and apertures.

- A lens should always be used between detector and sample for proper definition of the temperature sensing area and to increase the signal height.
- The detector signal should only be originated from the surface of the sample and not from any surrounding parts. Therefore, for an automatic adjustment of the temperature sensing area a vertical lens shift device, a so-called *ZoomOptics,* should be optionally available. The *ZoomOptics* should be software-controlled.

Light Flash

- The light flash source should be positioned beneath the sample and the distance between flash lamp and sample should be minimized and the mirror system should be optimized so that no additional wave guard is necessary.
- The flash source must have a variable energy of up to 10 J/pulse.
- Additional flash source energy reduction by an optional filter (for e.g., thin films; 25%, 50%, 75% and 100% of pulse energy) should be implemented. The filter wheel should be software controlled.
- The flash energy should be software controlled.
- The pulse width should be minimum 20 µs
- The flash pulse width must be adjustable from 10 µs to 1500 µs.
- The system must be equipped with a pulse mapping device to measure the actual pulse shape for each individual pulse (necessary for calculation of thermal diffusivity and specific heat,

Sample Position, Thermocouple, Automatic Sample Changer (ASC), Sample Holders

- The system shall be equipped with an automatic sample changer (ASC). It should be possible to measure of up to sixteen (16) samples with a diameter of 12.7 mm in the temperature range from -100°C to 500°C.
- The sample position within the furnace should not be changed during a measurement and thermocouple should be integrated in the furnace and should be as close as possible to the sample that is being tested.
- The system must allow measurements on round & square samples, with diameters/square sizes between 10 mm and 12.7 mm (with a tolerance of 0.0/-0.3 mm) & sample thicknesses between 0.01 mm and 6 mm, depending on the samples' thermal diffusivity. For highly conductive and thin materials no limitations of the sample thickness should be given.
- Sample holders made should not be made of alumina. The sample holders should be made of optically dense materials, e.g. stainless steel. Special sample holders for molten polymers, low viscosity liquids (e.g., water, possible viscosity shall be 0.2 to 10^{10} mPa*s)), pastes, powders, laminates and fibers as well as for measurements in-plate direction (in-plane) and for tests under mechanical pressure should be available. Special sample holder with low cost consumables (for e.g. resins during curing) should be available.
- The sample holders should have identification marks/numbers for each sample position for safe distinction between the 16 possible samples.
- For calibration and especially for c_p determination Pyroceram should be available among further reference materials.

Data Acquisition and Software

Data Acquisition

- For the detector channel the data acquisition system should allow acquisition rates of up to 2 MHz (0.5 μs). Two independent data acquisition systems should be built in. The data acquisition system must allow minimum 16,000 measurement points for the detector and pulse signal, each.
- For high-conducting and /or thin samples (especially for e.g., Al, Cu plate, thin foils) the minimum measurement time should be at least 1 ms ($t_{0.5}$= 100 … 200 μs).
- For low-conducting and/or thick samples (e.g., polymers, refractories, etc.) the maximum measurement time should be up to 120 s.
- The system must allow manual or fully automatic adjustment of measurement times and amplification (e.g., automatic measurement overnight).

Software

- The software shall operate the instrument in the fully automatic or manual mode.
- Standard and reference materials should be predefined and available with one mouse click.
- The software should include at least three (3) baseline corrections. It should be possible to modify and change the baseline correction in subsequent analysis.

- After start of the measurement the software should save the test results in the data base very instant of time. The analysis software should include minimum 15 different models for evaluation of the measured signals; each model can be combined with three different baseline corrections and with or without pulse correction.

- The software must have the capability of correcting the experimental data for the finite pulse width of the light flash and radial and facial heat loss simultaneously (based on a non-linear regression routine and an improved heat diffusion model (Cape-Lehman)).
- The software should have a model for correction of radiation heat transfer simultaneously to finite pulse and heat loss corrections (translucent samples).
- The software shall contain mathematical models for two- and three-layer samples as well as contact resistance calculation on the basis of a non-linear regression routine including heat loss and finite pulse correction.
- The software shall have the capability of calculating the thermal conductivity using measured thermal diffusivity data along with data of the specific heat and bulk density.
- The software should allow specific heat determination on the basis of a comparative method. It should be possible to carry out a c_p determination after the measurement – even without having c_p determination chosen before measurement start.

- All information (data files for individual samples at various temperatures) pertaining to a test should be kept in a database and therefore it should be possible to simultaneously load multiple measurements from that database in a single step.
- Software should take into account the penetration of the laser beam into the specimen according to McMasters (model)
- Software should take in to account the actual illuminated area by the laser beam and actual detection area of the IR –detector.

Computer :

Computer for operating the thermal conductivity instrument with Windows 10, core i5 processor 1TB HDD SATA & 4 GB RAM DDR4: Optial Drive, USB Key board Mouse Bundle, 17inch monitor or better should be supplied along with the instrument free of cost.