

Laser Flash Analysis – LFA

Method, Technique, Applications



Laser Flash Analysis

Due to the ever-increasing number of materials being used in high-temperature applications, knowledge of their thermophysical properties, especially thermal conductivity, is of paramount importance.

One of the most widely used methods for determining thermal conductivity is to measure the thermal diffusivity (a), specific heat (c_p) and density (ρ) as a function of temperature, and then to compute the thermal conductivity from these data. That is:
$$\lambda(T) = a(T) \cdot \rho(T) \cdot c_p(T).$$

By far the most popular and widely used instrument for measuring thermal diffusivity is the laser flash apparatus. It has been estimated that this technique is used in over 80% of thermal diffusivity measurements conducted worldwide. Compared with the direct measurement of thermal conductivity, the advantages of this non-contact, non-destructive method are simple sample geometry, easy sample preparation and small sample size, as well as applicability for a wide range of diffusivity values and excellent accuracy and reproducibility.

Also, because very little time is needed for a single measurement, a wide range of temperatures can be covered in a short period of time.

The completely enclosed system with its compact and space-saving design meets the highest safety requirements (laser class 1).

No additional safety precautions are necessary. The design (vertical alignment of laser output and IR detector) allows simple, horizontal insertion of the sample. Also, the short

distances involved minimize the loss of laser energy and energy impinging upon the IR detector. Measurements can be carried out in a static or dynamic oxidizing or inert gas atmosphere or under vacuum. Further, the evaluation of 2- and 3-layer samples and of the contact resistance between layers is possible.



Principle

The sample is mounted on a carrier system which is located in a furnace. After the sample reaches a predetermined temperature, a burst of energy emanating from a pulsed laser is absorbed on the front face of the sample, resulting in homogeneous heating. The relative temperature increase on the rear face of the sample is then measured as a function of time by an IR detector.

The thermal diffusivity is computed by the software using these time/relative temperature increase data.

For adiabatic conditions, a is determined by the simple equation

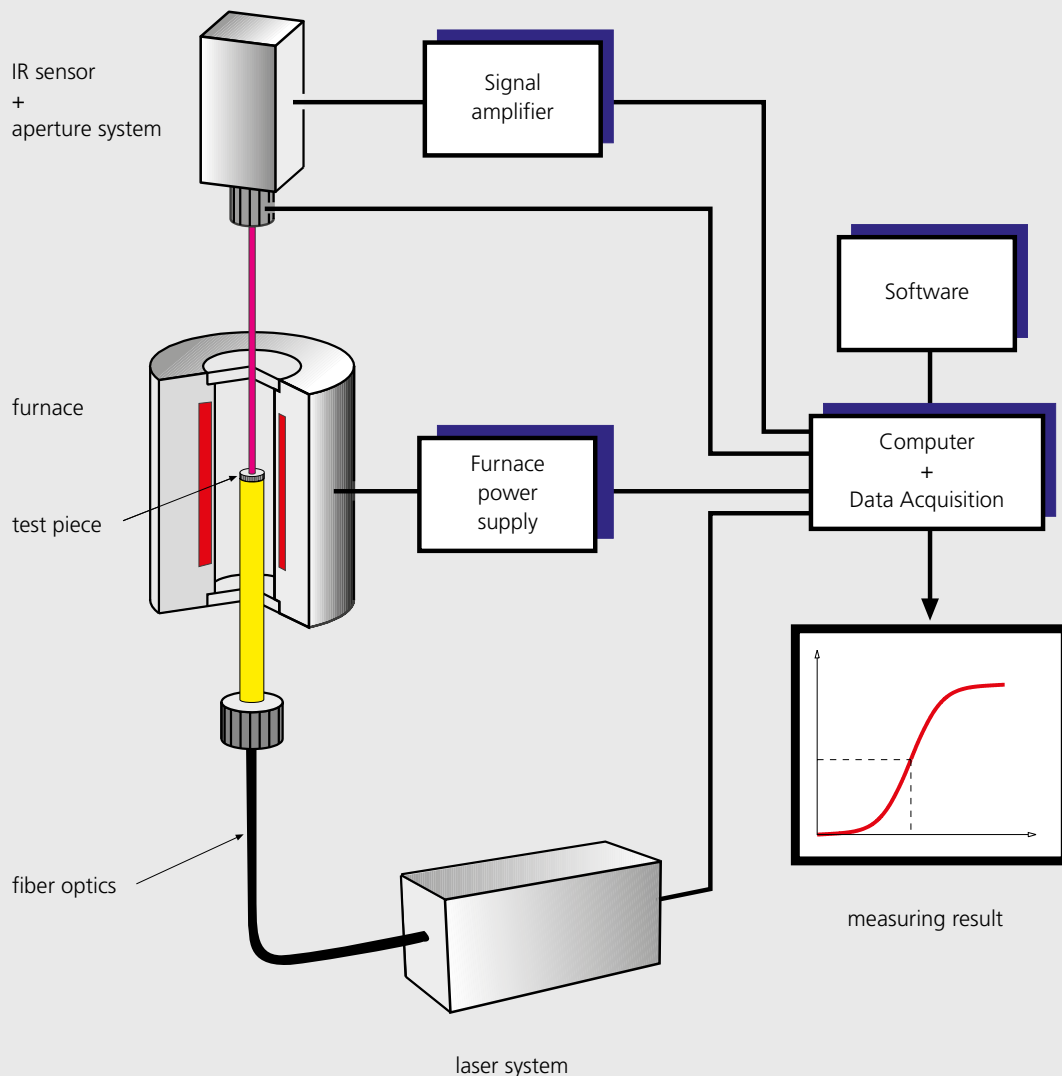
$$a = 0.1388 \frac{l^2}{t_{0.5}}$$

where

l = sample thickness

$t_{0.5}$ = time at 50% of the temperature increase.

The elegance of the method lies in the fact that the troublesome measurement of the absolute quantity of laser energy absorbed by the sample and of the resulting absolute temperature increase is replaced with a more accurate and direct measurement of time and relative temperature increase.



Hardware Concept

The LFA 427 consists of four essential components:

- Measuring unit with furnace, sample carrier and IR detector
- Controller for measuring unit
- Laser system connected via fiber optics
- Data acquisition system and computer

The vertically-constructed measuring unit is the primary component of the

system. The laser system is connected with the measurement part by a sheeted glass fiber. The beam emerges from the outlet of the fiber optics. A recipient block is located above the laser optics. The tube-shaped sample holder and its adjusting device are mounted on this block. A toggle quick seal permits a vacuum/gas-tight coupling between the recipient block and the furnace. The furnace system is raised and lowered

by means of a motor-driven hoist.

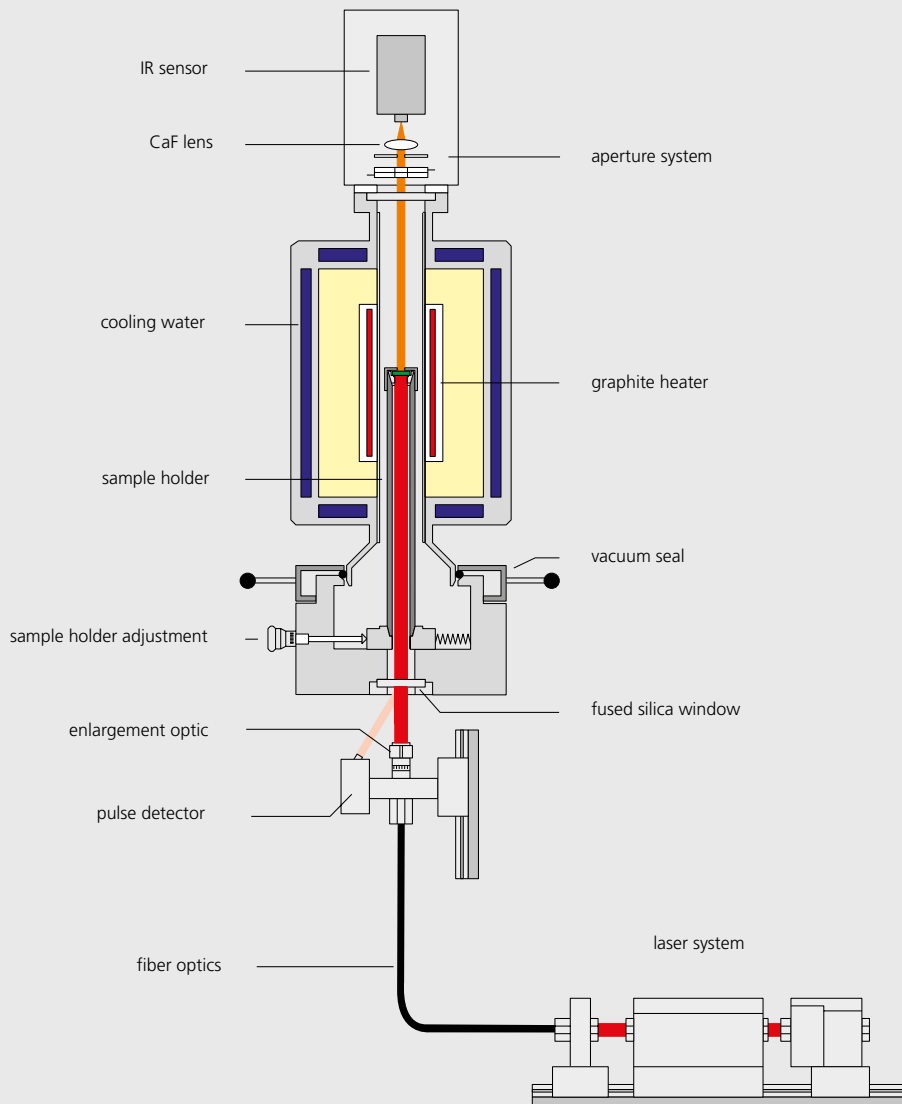
A solid-state laser produces a pulse with the energy which is required for the flash method. The emission wave length of 1064 nm is in the infrared range. The pulse width of the laser is variable.

A thermocouple measures the absolute temperature of the sample (-120°C to 2000°C). This thermocouple is mounted laterally on the sample carrier tube at the same height as the sample. Different thermocouples can be used, depending on the temperature range of interest. At the 2800°C-system the same thermocouple is replaced by a wide range pyrometer (RT to 2800°C).

An IR detector measures the temperature increase on the rear face of the sample. The indium antimonide or mercury cadmium telluride detectors are liquid nitrogen cooled. Further features of this detectors are the CaF lens, which also acts as a filter for the laser light, and a motor-driven aperture system.

With regard to safety requirements, this is a "laser class 1" instrument. This means that the apparatus need not be housed in a separate room, nor do laser safety glasses need to be worn by the operator. These requirements have been eliminated with the completely enclosed design of the measuring unit and with a safety system electronically connected to both the hoist and front door of the cabinet. This safety system closes the shutter of the laser system immediately if the hoist is moved or the front door opened, thus preventing release of the laser pulse.

Hardware Schematic (2000°C furnace)



Furnaces

The LFA 427 can be equipped with different furnaces. A liquid nitrogen cooled furnace allows tests between -120°C and 400°C. A forced air-cooled SiC furnace enables tests between room temperature and 1575°C.

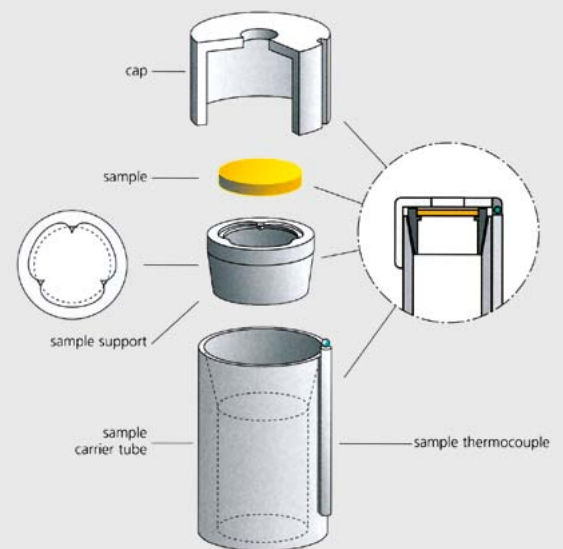
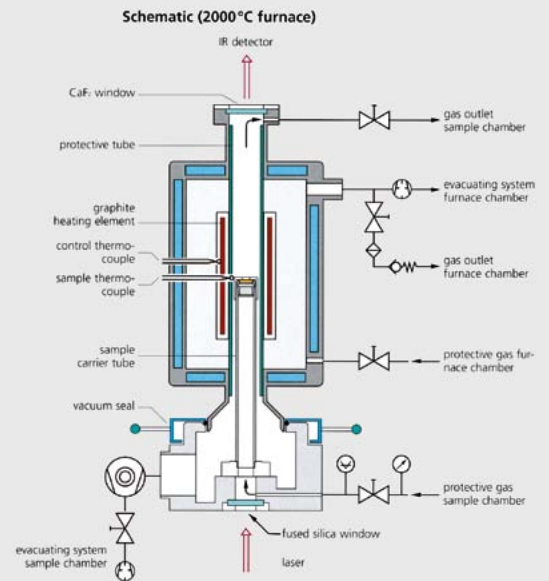
The 2000°C/2800°C furnace consists of a graphite heating element and graphite insulation mounted in a water-cooled housing. The furnace and the sample chamber are separated by a glass carbon tube (2000°C) or an Al₂O₃ tube (1700°C max. temperature). The Al₂O₃ tube allows measurements to be conducted under high vacuum or in static or dynamic inert gas or oxidizing atmospheres, while the glass carbon tube allows measurement under high vacuum or in static or dynamic inert gas atmospheres.

Sample Holder Systems

The vertical design of the measuring unit allows very easy sample handling. The sample is in a horizontally stable, well-defined position. Once the furnace has been raised and swung to the side, the sample is directly accessible and can be easily inserted or removed.

An Al₂O₃ or graphite sample carrier tube mounted in a metallic adjusting socket carries the sample support, sample and sample cap. The sample support is mounted directly in the cone-shaped opening of the carrier tube. The sample support holds and centers the sample on three "teeth". This design minimizes the contact between the sample and sample support, thereby reducing heat loss and allowing uniform laser irradiation of almost the entire sample surface. The inside diameter of the sample

The different furnaces can easily be exchanged by the operator. The top and bottom of the sample chamber are sealed by calcium fluoride and fused silica windows, respectively. The IR detector, which is mounted directly on top of the furnace, has "visual" contact with the back face of the sample, allowing measurement of the temperature increase. The end of the fiber optics, located directly under the furnace, fires through the fused silica window, resulting in heating of the front face of the sample. The windows can be easily removed for cleaning.



Sample holder system

support is constructed as a limiting diaphragm below the sample and corresponds exactly to the diameter of the sample. The impinging laser beam with a larger diameter is masked exactly to the sample diameter.

A cap, which is placed over both the sample and sample holder, prevents the laser energy from impinging directly on the detector. This prevents detector damage as well as signal disturbance.

Sample holders for smaller samples, square samples, laminates, liquid metals, slags or fibers can be employed at the sample support.

Software

The LFA 427 comes equipped with a 32-bit-Windows® operating system, especially tailored to the needs of our users. It combines easy handling and complex evaluation routines, thus offering a solution to almost every problem the user might face.

More than 15 different evaluation models available for the user, have been developed with leading experts from science and industry and correspond to current requirements and state-of-the-art technology.

Software Features

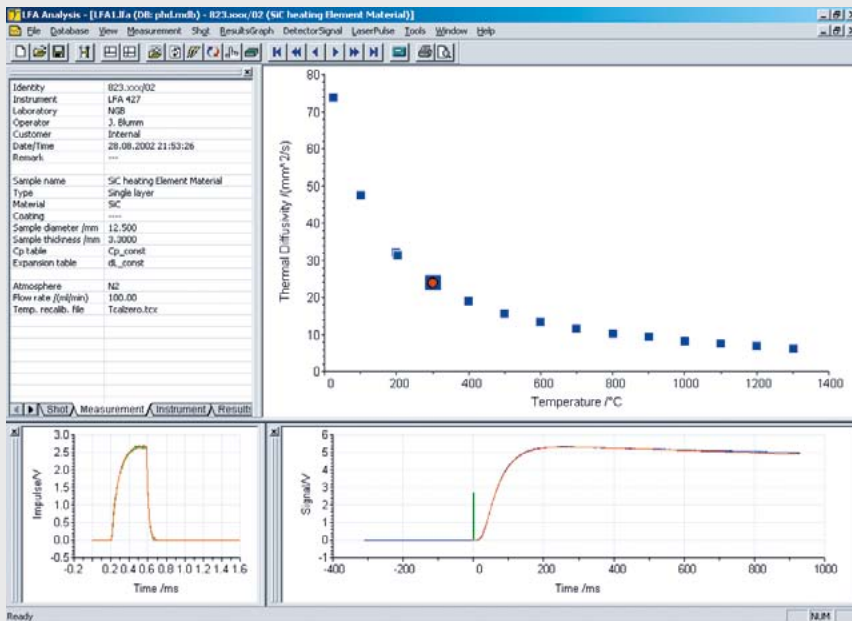
- 32-bit-Windows® operating system: fully compatible with other programs that run with Windows® operating system
- Multitasking: simultaneous measurement and evaluation
- Full network compatibility
- Easy printout and export of measuring curves and data (ASCII)
- Selectable screen design by means of docking windows
- Multi-moduling: operation of several different instruments with one computer
- Integrated data base

Evaluation Task

- Presentation of an individual response curve, the entire result as well as test parameters and measured values in one presentation
- Free input or import of density and specific heat values for determination of thermal conductivity
- Simultaneous presentation of thermal diffusivity and conductivity data in one plot
- Storage and restoration at any point of the analysis
- Presentation and new evaluation of data from previous measurements

Measuring Task

- Easy and user-friendly input of test parameters
- Free selection of temperature programs with heating, isothermal and cooling segments
- Temperature calibration: a unique tool for calibration of the sample thermocouple by the user is introduced
- Manual or automatic optimization of the system parameters (measuring time, amplification, etc.)
- Automatic recording and storage of the laser pulse for each shot for an optimum finite pulse correction
- Automatic evaluation of the measurement after each shot with one evaluation models



Perfect fit of the response curve (blue) due to the Cape-Lehmann model (red curve)

Evaluation Models

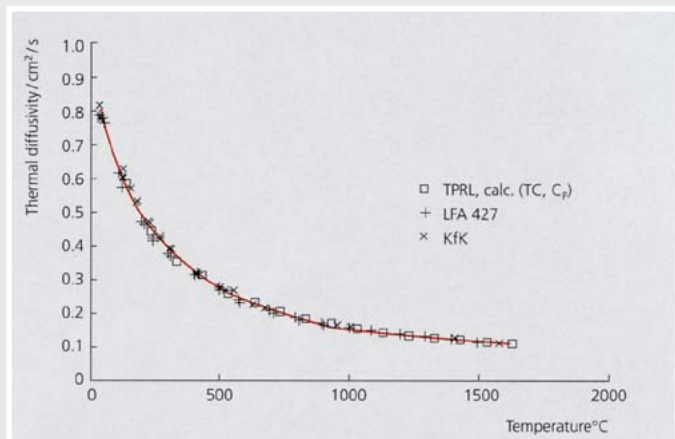
- Accurate finite pulse correction based on the corresponding laser pulse
- Standard heat-loss corrections: correction models according to Cowan, Clark and Taylor, and Degiovanni known from literature, are integrated
- Cowan-Fit: non-linear regression based on the original publication by Cowan (optionally with or without finite pulse correction)
- Cape-Lehmann-model is improved. Non-linear regression with consideration of radial and facial heat losses (optionally with or without finite pulse correction)
- Correction of radiation effects: a new model that considers radiation effects is integrated for accurate analysis of tests on oxide ceramics or glasses (simultaneously with heat-loss and finite pulse corrections)
- 2- or 3-layer systems: analysis of multi-layer systems with consideration of the heat loss based on non-linear regression (optionally with or without finite pulse corrections)
- Contact resistance: determination of the contact resistance in a 2-layer system
- Model wizard: simultaneous evaluation of a response curve with several models; determination of the optimum model using statistic criteria
- Determination of specific heat by means of a comparative method

Applications

Comparative Measurements

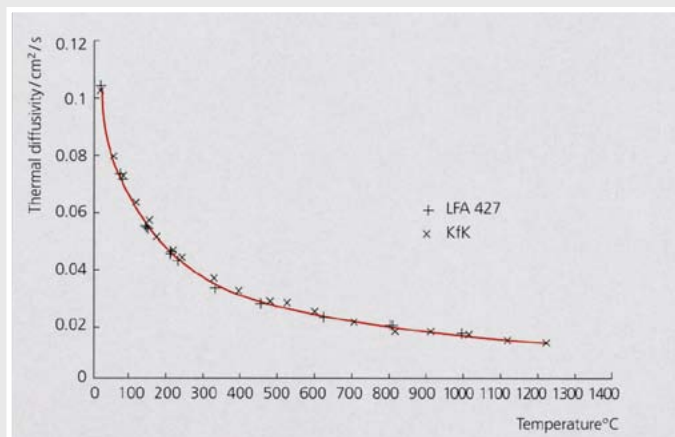
POCO graphite

This figure shows the results (LFA 427, FZK*) of thermal diffusivity measurements on POCO graphite compared to values calculated from measured thermal conductivity and specific heat data (TPRL**).



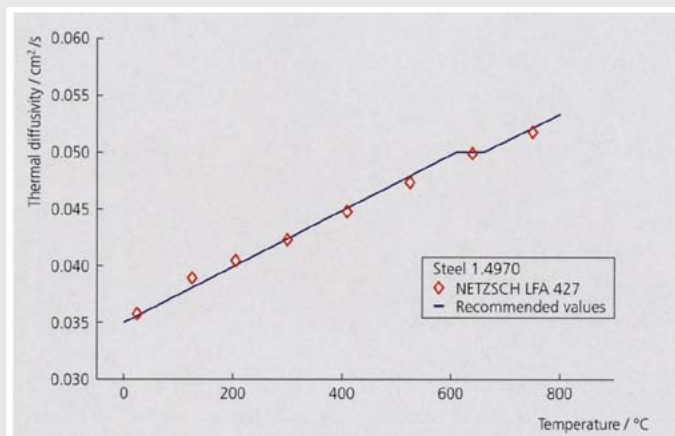
Bio-Al₂O₃

Test results (LFA 427, FZK*) are shown for a polycrystalline Bio-Al₂O₃ sample. Both faces were coated with a graphite emulsion.



1.4970 Steel

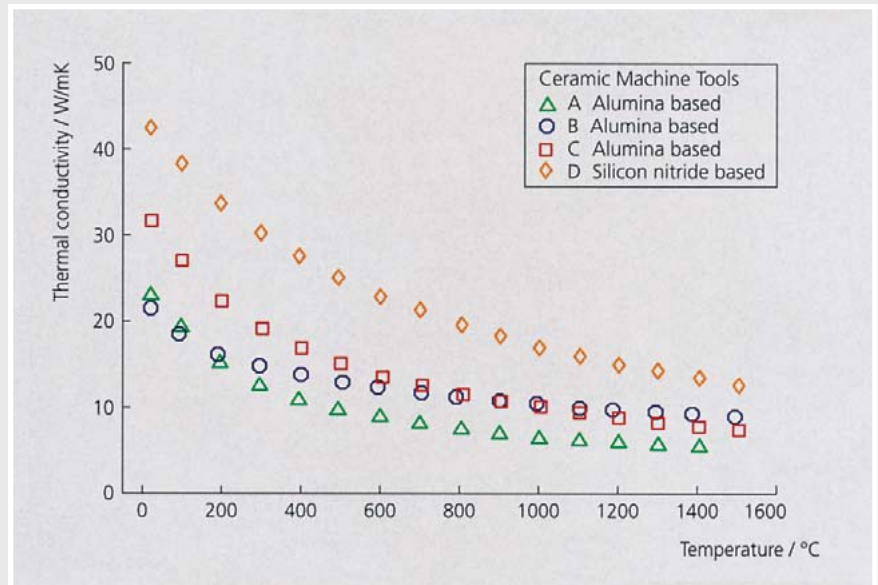
Shown here is the comparison of thermal diffusivity values measured with the LFA 427 and those recommended by the "Arbeitskreis Thermophysik" in Germany. The maximum deviation between the two sets of data occurs at 125°C and is only approximately 2.2%.



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** Thermophysical Properties Research Laboratory, Purdue University, USA

Ceramic Machine Tools

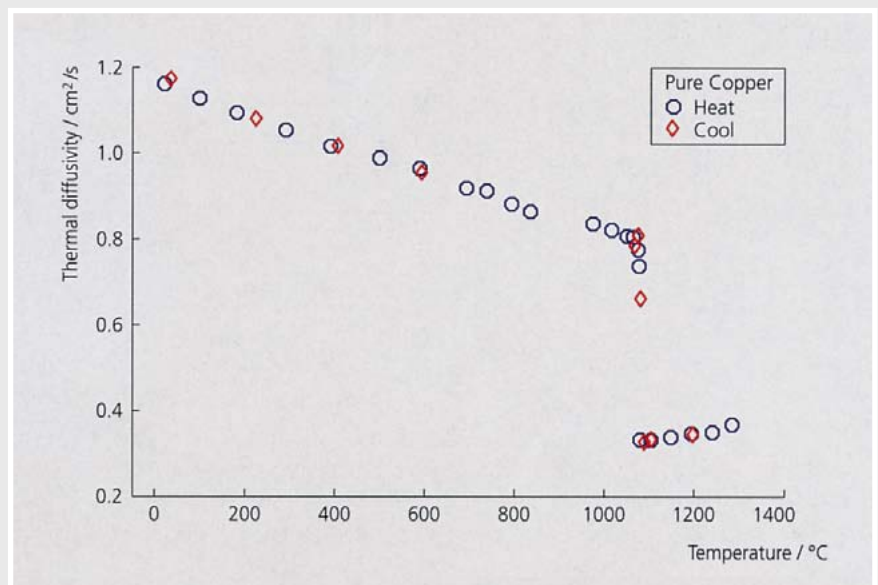
Due to the increased machining speeds and better wear and chemical resistance, ceramics have replaced metals in many applications. However, since ceramics generally have lower thermal shock resistance, the temperature gradients which develop in the tools during use are of paramount importance. As a result, the thermal diffusivity (\Rightarrow thermal conductivity) of these materials is a primary design consideration. This figure shows the thermal conductivity of four ceramic machine tools. Ceramics A, B and C are all Al_2O_3 -based, each with different secondary components, while sample D is Si_3N_4 -based. The higher thermal conductivity of the Si_3N_4 allows it to be liquid cooled, while samples A and B must be used without liquid cooling. Sample C can be liquid cooled at lower temperatures.



* The thermal diffusivity and specific heat for all application examples were measured with the LFA 427 and DSC 404, respectively. The thermal conductivity values were computed from the thermal diffusivity, specific heat and bulk density data.

Pure Copper

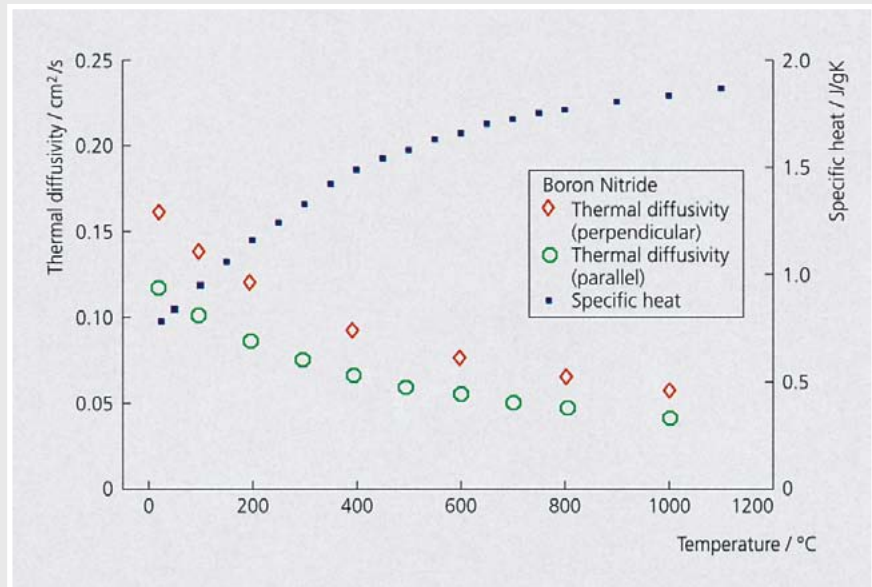
The thermal diffusivity of pure copper was measured for both the heating and cooling cycles. The large change in the thermal diffusivity at approximately 1080°C is due primarily to the change in the electronic component of the thermal conductivity upon melting or solidification. The fact that there is almost no difference in the thermal diffusivity values between the heating and cooling cycles indicates that no significant microstructural changes occurred. The measured values of the thermal diffusivity for both the solid and liquid regions deviated from those found in the literature by less than 2.5%. Note that the change in thermal diffusivity provides a good temperature calibration point for the LFA (melting point of pure copper = 1083°C).



Applications

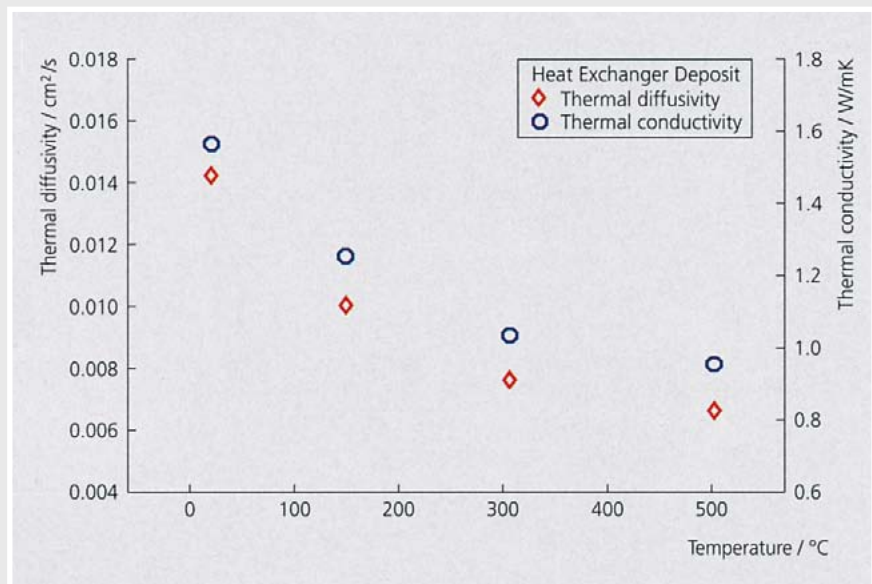
Boron Nitride

Pure hexagonal boron nitride is a soft material (similar to graphite) and maintains its excellent insulating properties to temperatures above 2000°C. It is therefore used as a high-temperature insulating material. In addition, boron nitride has an excellent thermal shock resistance. This figure depicts the specific heat and thermal diffusivity of hot pressed boron nitride (bulk density $\approx 2.1 \text{ g/cm}^3$). Because of its structure, the thermal diffusivity parallel to the direction of press is lower than that in the perpendicular direction. This, of course, is the expected behavior.



Heat Exchanger Deposit

With time, heat-transfer surfaces in heat exchangers can become coated with various types of deposits. These deposits result in an additional resistance to heat flow, thereby lowering performance. This effect is usually referred to as fouling. In order to quantify the impact of fouling on heat exchanger efficiency, the thermal diffusivity (\Rightarrow thermal conductivity) of the deposits must be determined. This figure depicts the measured temperature-dependent thermal diffusivity and calculated thermal conductivity of a heat exchanger deposit.



Technical Data

Temperature ranges	-120°C to 400°C 25°C to 1575°C 25°C to 2000°C 25°C to 2800°C
Diffusivity range	0.01 mm ² /s to 1000 mm ² /s
Standard sample dimensions	0.1 mm to 6.0 mm thick, 6.0 mm to 12.7 mm diameter up to 20.0 mm diameter (special version)
Standard sample holder system	Al ₂ O ₃ (max. 1700°C) graphite (max. 2800°C)
Furnaces	2800°C: graphite heating element with water cooled housing 2000°C: graphite heating element with water cooled housing 1575°C: silicon carbide heating element 400°C: resistance heater with LN ₂ -cooling
Test atmospheres	vacuum: 10 ⁻⁵ mbar (turbomolecular pump) static/dynamic: protective gas (Ar, He, etc.), oxidizing atmosphere
Laser	Nd: YAG (neodymium: yttrium aluminum garnet) power max.: 20 Joules/pulse pulse width: variable wave length: 1064 nm
IR detectors	InSb (Indium-Antimonide) MCT (Mercury Cadmium Telluride) with liquid nitrogen cooling
Sample holders	standard 12.7 mm diameter standard 10 mm diameter standard 6 mm diameter square samples: 10 mm x 10 mm liquid materials slags fibers/powders laminates/in-plane mechanical pressure

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