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OnSet 15

News, Facts and Professional Solutions for Thermal Analysis

In this edition:

NETZSCH GABO Instruments – The DMTA-Expert from Ahlden as a New Member of the NETZSCH-Group

Dr. Herbert Mucha, Application Support, NGI Ahlden

Fig. 1. The NGI team along with some guests from NGB in front of NGI's main building in Ahlden, Germany

On July 9, 2015, Gabo QUALIMETER, the renowned manufacturer of high-load DMA instruments (also called DMTA), has become an independent subsidiary of the NETZSCH Analyzing & Testing Business Unit. Their new name, NETZSCH GABO Instruments – NGI for short – has already become established in the company.

The NGI and NETZSCH Analyzing & Testing business segments are quite complementary to one another and offer significant advantages to both: NGI is now in the position to provide quick local service almost anywhere in the world – which is often a central factor in industrial customers' purchasing decisions – while NETZSCH has become the

LFA 467 *HT HyperFlash*® – First LFA with Xenon Light Source to 1250°C

Dr. Elisabeth Kapsch, Technical & Scientific Communication

Fig. 1. Front view of the LFA 467 *HT HyperFlash*®

In 2013, we introduced a new design for a laser/light flash apparatus with the LFA 467 *HyperFlash*®. The design then established has now become the basis for our new high-temperature LFA 467 *HT HyperFlash*®, which has the world's first xenon flash lamp capable of heating to temperatures of 1250°C (figure 1). Due to the innovative light source, the footprint is comparable to that of an LFA 467 *HyperFlash*®. This light source, which sets itself apart by means of its long lifetime, requires no categorization into a laser class.

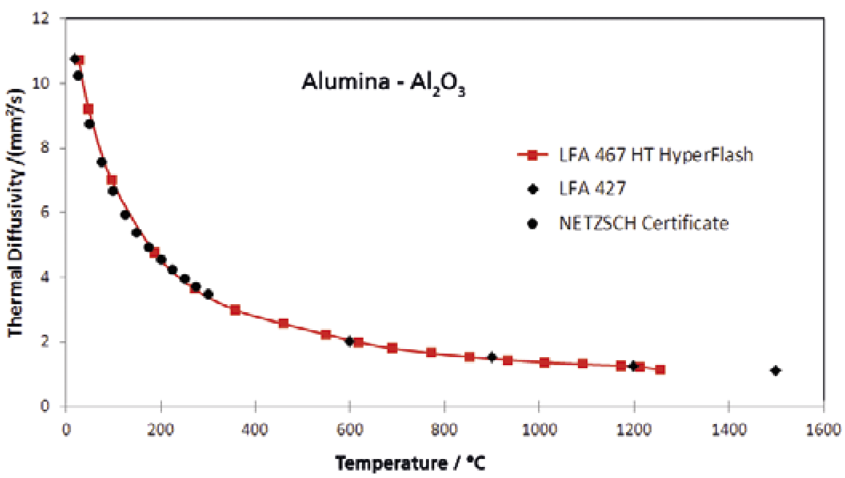


Fig. 2. Measurement on alumina up to 1250°C with the use of a xenon flash lamp

1250°C with a Minimal Footprint and Defined Atmosphere

A single furnace with an integrated sample changer covers the entire temperature range while still allowing the small footprint for which the LFA 467 *HyperFlash*® series is known (figure 2). The vacuum-tight platinum furnace (up to 10^{-5} mbar) allows for heating rates up to 50 K/min. An efficient internal water-cooling circuit protects the surrounding instrument components from overheating, thereby also minimizing the liquid nitrogen consumption of the IR detector. An automatically regulated pump device for evacuation prior to each measurement and built-in mass flow controllers (MFCs) ensure that measurement conditions are well defined. Connections for external pump devices are available, allowing for pure atmospheres during the measurement (e.g., for oxygen-sensitive samples).

4 Samples – 4 Thermocouples

The automatic sample changer (ASC) can accommodate up to four samples and, together with the fast heating rates, thus boosts sample throughput (figure 3).

Fig. 3. Furnace with integrated automatic sample changer

In contrast with conventional systems, each of the sample positions in the ASC of an LFA 467 *HT HyperFlash®* is equipped with its own thermocouple. This makes it possible to avoid the occurrence of any temperature deviations between the sample and temperature measurement position, in order to individually control each sample position. The ASC allows for round (12.7 mm) and square (10 mm) sample geometries.

ZoomOptics for a Defined View of the Sample Surface

As with the LFA 467 *HyperFlash®*, a stepper motor actuated lens between the sample and detector optimizes the detector's field of view via software control. This helps prevent measuring artifacts attributable to the sample's immediate surroundings (e.g., masks or aperture stops). The precision of the test results is thus greatly improved. At the same time, there is an improvement in the signal-to-noise ratio, which is associated with a reduced flash power and therefore also directly affects sample heating. *ZoomOptics* is also especially advantageous for measurements with different sample diameters.

Effortless

The InSb detector can be equipped with a refill system for liquid nitrogen, whereby even with multiple temperature steps, many sample tests can be carried out without operator invention.

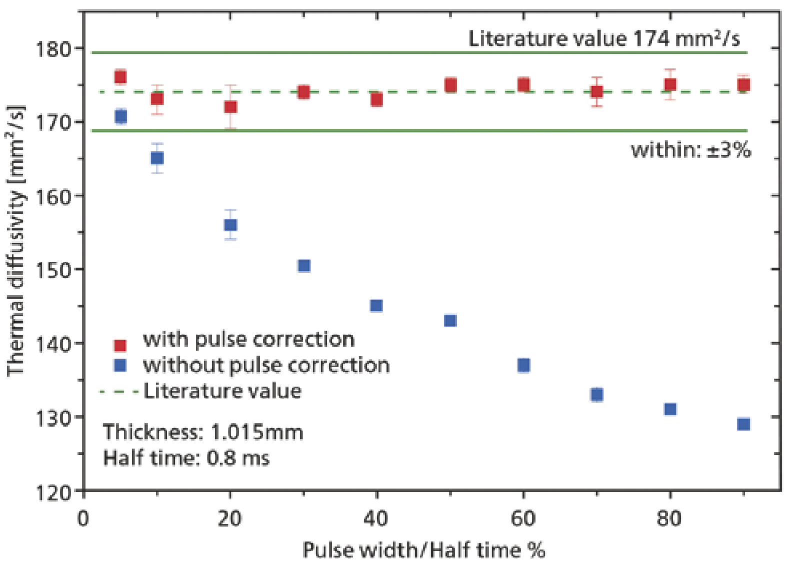
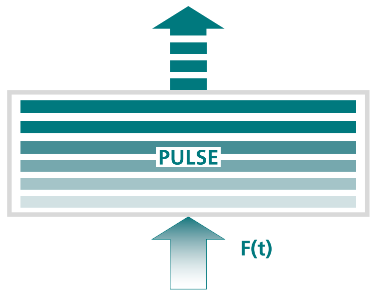


Fig. 4. Measurement on a thin silver film

Thin and Highly Conductive Materials Require High Data Acquisition and Low Pulse Widths



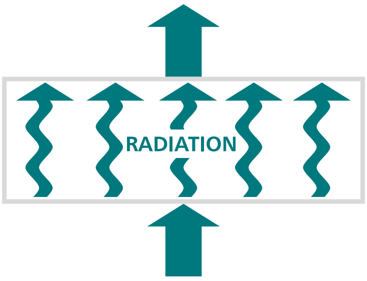
Finite pulse correction

The high data acquisition rate of 2 MHz applies to both the IR detector and the pulse mapping channels. Thereby, highly conductive and/or thin materials requiring short test times can be reliably tested. Pivotal in evaluating the thermal diffusivity of these materials is the recording of the real laser pulse of each individual measurement along with its mathematical description. The unique pulse mapping (patent no. US 7038209, US 20040079886, DE 10242741) enables finite pulse correction and an improved determination of thermal diffusivity (a) and specific heat capacity (c_p). This correction method is implemented in the standard software of the LFA 467 *HyperFlash®* series.

Figure 4 illustrates the influence of an optimized pulse correction with the example of a measurement on a 1.015-mm thick silver film at 25°C. Precise measurement results (within ± 3% of the literature value) can only be obtained when an intelligent pulse correction is used.

Translucent Samples Require their Own Radiation Model

For translucent samples, the light pulse causes an immediate temperature increase on the rear side of the specimen; this cannot be precisely described by conventional



Radiation/translucent model

models. Only by using a special model for radiation correction which takes the ballistic heat transfer by radiation into account can a correct adjustment (figure 5, red curve) to the detector signal (figure 5, blue curve) be achieved. The measurement on a glass ceramic demonstrates the effectiveness of this radiation model. The improved fit results in a considerably lower thermal diffusivity value (0.877 mm^2/s, right plot in figure 5) compared to the poorer fit (0.974 mm^2/s, left plot in figure 5) based on a conventional model. This radiation model – like many others – is also standard in the LFA *Proteus*® software.

Conclusion

The LFA 467 *HT HyperFlash*® is the world's first LFA apparatus covering a temperature range of 1250°C with a flash lamp. Innovations previously introduced with the *HyperFlash*® series (see On**Set**[12]), such as *ZoomOptics* (lens system) and the small footprint, are also a part of this instrument model.
The ASC has its own thermocouple for each sample position, thus minimizing differences between the sample and temperature measuring position. The LN_2 consumption of the detector is reduced by means of integrated water cooling. With an additional refill system, measurements can be carried out around the clock.

The LFA *Proteus*® software includes, as standard, highly developed correction models and mathematical operations which – together with the sophisticated hardware – provide the basis for precise measurement results. This makes the LFA *HT HyperFlash*® to a successful instrument for investigating the thermophysical properties across a broad temperature range.

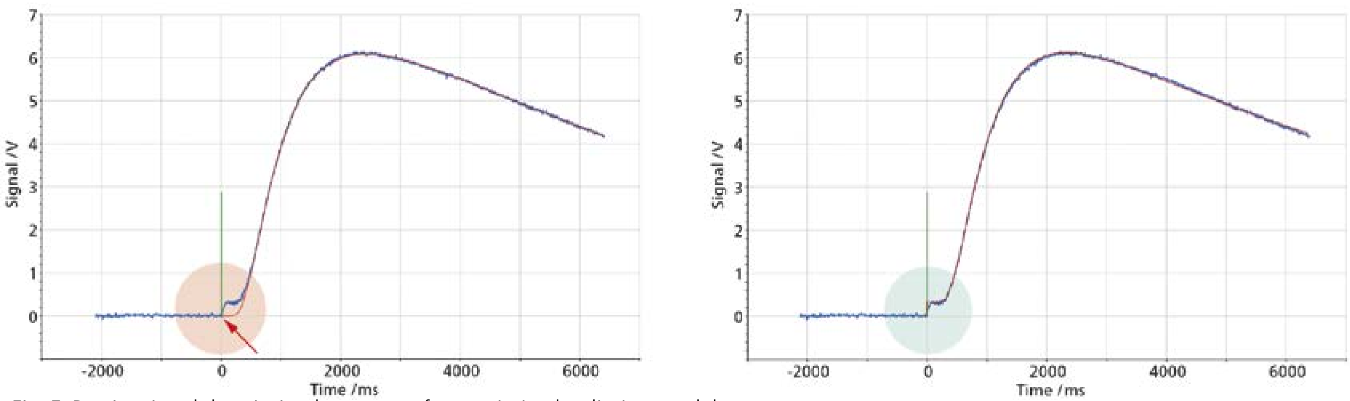


Fig. 5. Precise signal description by means of an optimized radiation model
left: conventional heat-loss model (standard)): 0.974 mm^2/s; right: radiation model: 0.877 mm^2/s