APPLICATION**NOTE**

# LFA 467 *HT HyperFlash*®: New Sample Holder Dedicated to Liquid Metals

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## Introduction

For LFA measurements, a defined sample thickness is required. The thermal diffusivity (a) is proportional to the square of the sample thickness (d): $a \sim d^2$. This demands high precision to obtain the exact thickness value. In addition, the heat flow through the outer container walls in the axial direction can be critical for sample holders for liquids. Furthermore, measurements on metal melts can destroy the sample holder. In order to address these critical issues, a new sample holder specifically for liquid metals was developed (figure 1). The special design, with some parts made of stainless steel or SiC and inner parts made of sapphire, allows for measurements with excellent IR-detector signals and therefore high precision. The metal is placed in a sapphire crucible which is closed with a sapphire lid on top. The defined sample thickness in the melt is realized by placing additional mass on top of the sapphire lid. This ensures flexible positioning of the lid in terms of height and prevents any damage to the sapphire part resulting from axial thermal expansion of the metal.

## Test Conditions

- Material: Aluminum alloy
- Instruments: LFA 467 *HT HyperFlash*®/DSC 404 ***F1*** *Pegaus*®
- Temperature range: 450°C → 750°C → 450°C
- Sample holder: For liquids and metals; made of sapphire; in SiC version, stainless steel (up to 750°C) and SiC version (up to 1250°C)
- Temperature range: 450°C → 750°C → 450°C
- Sample thickness: 1.5 mm
- Sample surface preparation: Thin graphite coating

**1** Design of the new sample holder for liquid metals; stainless steel (order no. LFA46700B96.040-00) and SiC version (LFA46700B96-041-00)

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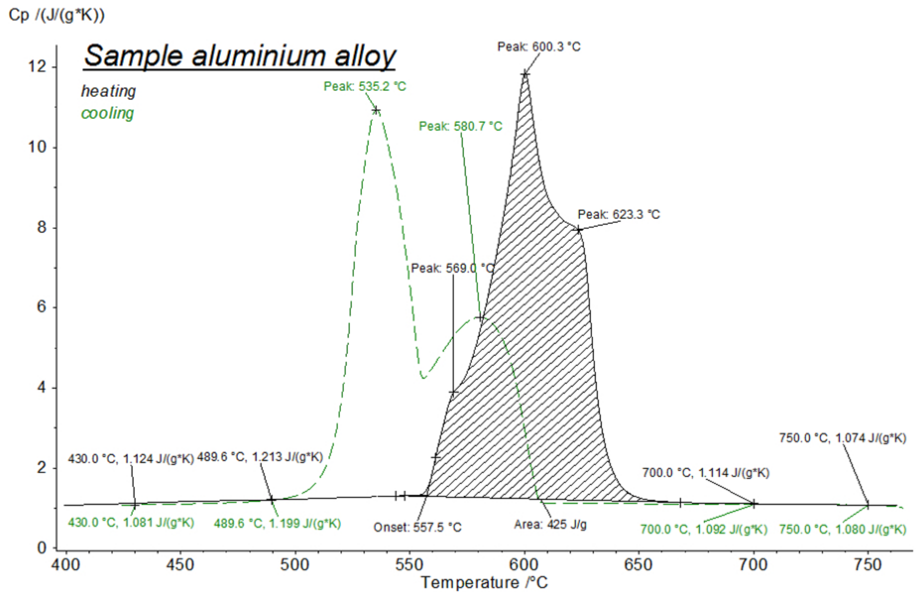


**2** Specific heat capacity including energetic effects of aluminum alloy during heating (solid black line) and cooling (dashed green line; DSC measurement).

## Measurement Results

The suitability of the new sample holder for liquids in conjunction with the LFA 467 *HT* was checked by means of a series of measurements on an aluminum alloy. Prior to the LFA test, additional DSC measurements were conducted. Figure 2 depicts the phase transition during heating and cooling in the DSC. During heating (black curve), multi-step melting of the alloy starts at 558°C (onset, solidus temperature) with peak temperatures at 569°C and 600°C. The last step is finished at 623°C (liquidus temperature). A slight sub-cooling effect can be seen in the cooling cycle (dashed green line). The crystallizing process starts between 610°C and 600°C, approximately 10–15 K below the liquidus temperature determined during heating. The crystallization ends at 535°C.

Depicted in figure 3 is the thermal diffusivity of the aluminum alloy during heating and cooling (LFA measurements). The values during melting and crystallization are in very good agreement, which indicates that the IR-detector has excellent signal stability and that conditions are stable both within and outside of the phase transitions (e.g., constant thickness of liquid/solid metal film). The solidus temperature is detected between 550°C and 575°C (by comparison, DSC: 558°C) and the liquidus temperature between 600°C and 625°C (by comparison, DSC: 623°C). The good agreement between the two independent instruments demonstrates the high temperature accuracy of the LFA 467 *HT*.

The calculation of thermal conductivity λ(T) is based on following equation:

$$\lambda(T) = \rho(T) \cdot c_p(T) \cdot \alpha(T)$$

where $\rho$ = density
$\alpha$ = thermal diffusivity
$c_p$ = specific heat capacity

The density, ρ, can be determined at room temperature by volume and mass. For precise results, a dilatometer can be used to consider for the thermal expansion and density change depending on temperature. The measured/calculated $c_p{}^*(T)$ DSC curves contain the contribution of the phase change enthalpies $\Delta h_{phase}$ and can be described as:

$$c_p{}^*\, dT = c_p\, dT + dh_{phase}$$

In order to get the "true" specific heat capacity $c_p(T)$ which is needed for the calculation of thermal conductivity, the phase change enthalpy must be subtracted:

$$c_p\, dT = c_p{}^*\, dT - dh_{phase}$$

That will usually be done by linear interpolation over the range of phase transition.

Figure 4 presents the thermophysical properties of the aluminum alloy including the calculated thermal conductivity for the solid-liquid phase transition.

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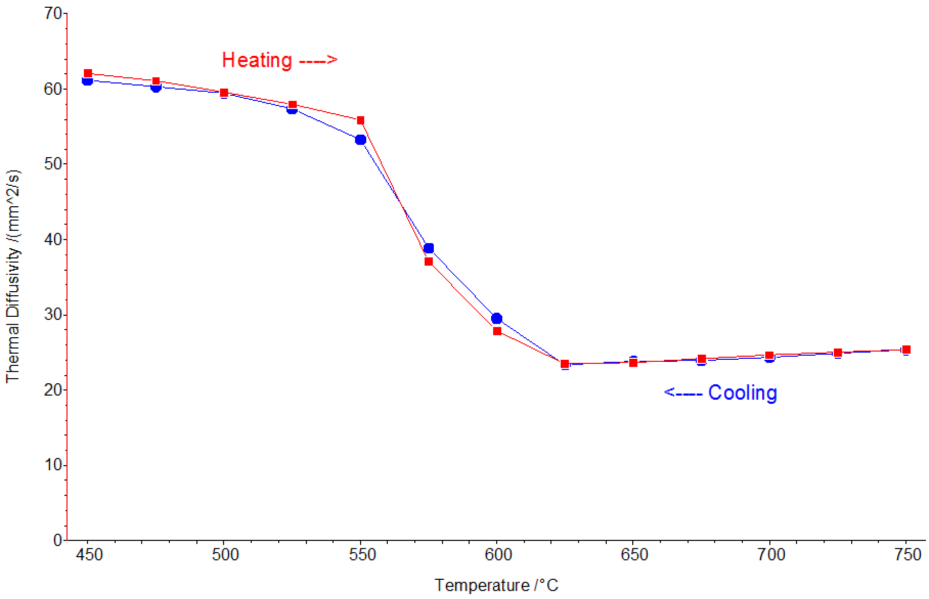
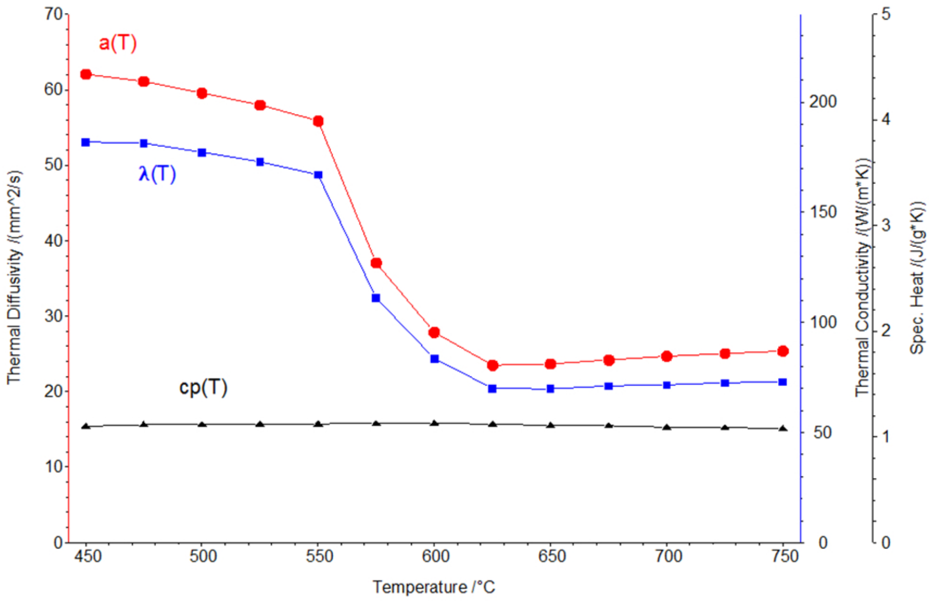



**3** Thermal diffusivity of aluminum alloy for the phase transitions solid → liquid and liquid → solid.




**4** Thermophysical properties of aluminum alloy for the phase transition solid → liquid.


## Summary

NETZSCH developed a new sample holder for liquid metals for the LFA 467 *HT HyperFlash*® which can be delivered in two versions, usable up to 750°C and 1250°C, respectively. Measurements on a liquid aluminum alloy clearly demonstrate the high reproducibility of the results during heating (melting) and cooling (crystallization).

The special design of the sample holder ensures constant sample thickness during the melt. At the same time, it prevents mechanical pressure on sapphire parts resulting from thermal expansion. Thanks to the excellent signal stability, high precision with low scattering was achieved. Furthermore, good agreement with DSC results was obtained and the detected phase transition temperatures were all in the expected range.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