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

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

Introduction

For LFA measurements, a defined sample thickness is required because 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, it should be considered that measurements on metals into the melt could easily destroy the sample holder.

In order to address these critical issues, NETZSCH developed a new sample holder for measurements on metals which need to be carried out at temperatures above the melting point (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. Measurements can be performed up to 750°C in the sapphire/stainless steel combination and up to 1250°C in the sapphire/SiC combination.

The sample (i.e., 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 an 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.

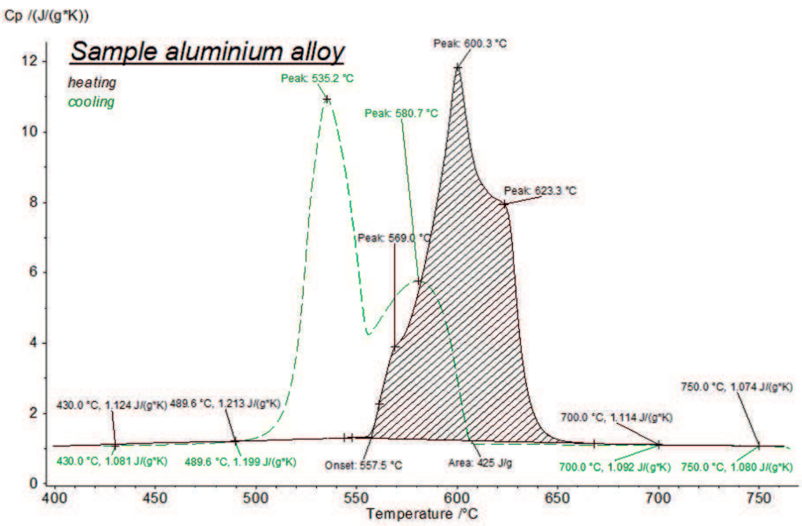

Fig. 2. Apparent specific heat capacity including energetic effects of an aluminum alloy during heating (solid black line) and cooling (dashed green line; DSC measurement)

Test Conditions

For the determination of the specific heat capacity (c_p) and thermal diffusivity (a), an aluminum alloy was tested in the temperature range between 450°C and 750°C using the LFA 467 *HT HyperFlash*® and DSC 404 ***F1*** *Pegasus*®. The sample thickness amounted to 1.5 mm; the sample surface preparation included a graphite coating. The new sample holder for liquids in the sapphire and stainless steel version was used.

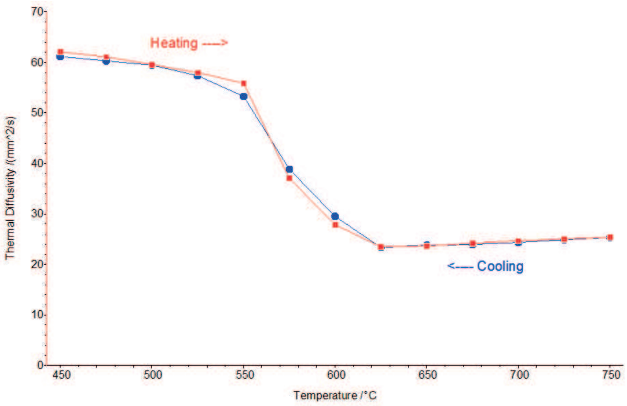

Fig. 3. Thermal diffusivity of the tested aluminum alloy between 450°C and 750°C; phase transitions solid → liquid → solid during heating (red curve) and cooling (blue curve)

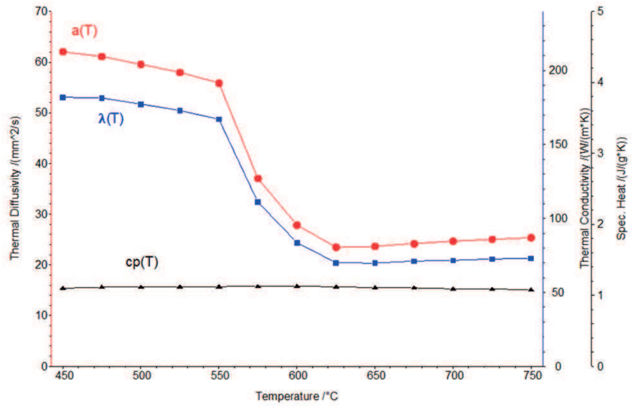

Fig. 4. Thermal diffusivity (red cuve), thermal conductivity (blue curve) and specific heat capacity (black curve) for the phase transition solid → liquid

Measurement Results

The suitability of the new LFA sample holder for liquids was checked by means of a series of measurements on the aluminum alloy with additional DSC measurements prior to the LFA tests. Figure 2 depicts the phase transition during heating and cooling detected 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). The DSC records a slight sub-cooling effect in the cooling curve (dashed green line). The crystallizing process starts between 610°C and 600°C, approximately 10 to 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 crystal-lization 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 methods demonstrates the high temperature accuracy of the LFA 467 *HT HyperFlash*®.

Figure 4 presents the thermophysical properties of the aluminum alloy including the calculated thermal conductivity (λ) and specific heat capacity (c_p) for the solid-liquid phase transition. The determination of the "true" specific heat capacity is based on the DSC measurement, under subtraction of the phase change enthalpy:

$$c_p\,dT = c_p \cdot dT - dh_{phase}$$

Summary

For the NETZSCH LFA 467 *HT HyperFlash*®, a new sample holder for liquids is available in two versions; one can be used up to 750°C and the other up to 1250°C. 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. The excellent signal stability is expressed in the high precision with low scatter. Additional DSC measurements confirm the LFA test results.

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