

APPLICATION NOTE

Molten Metals – LFA

LFA 467 HT HyperFlash®: Sample Holder for Measurement on Molten Metals

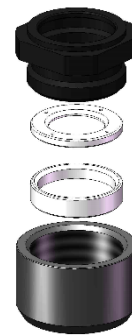
Anne Lichtinger

Introduction

Determination of the thermal diffusivity, α , by means of LFA requires knowledge of the sample thickness, d , since the thermal diffusivity is proportional to the square thickness. For measurement of molten metals by LFA, it must therefore be ensured that the sample thickness does not change during the measurement

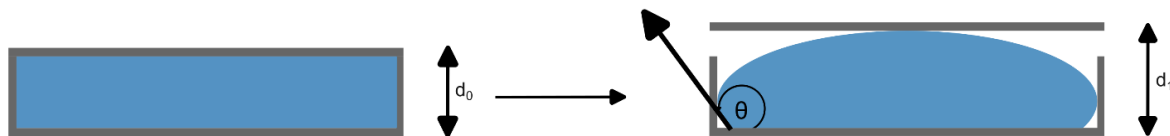
The sample holder for molten metals made of SiC (up to max. 1250°C) can be used for such measurement [1]. It consists of a sapphire crucible, into which the metal is placed and sealed with a sapphire lid [1]. Some molten metals have high surface or interfacial tension, γ , as exemplified by the high value of γ_{Cu} ($T=1058\text{ °C}$)= 1304 mN/m in copper [2]. This high surface tension causes metals to form droplets during melting (figure 1). This may expand the sample thickness (from d_0 to d_{0i}), resulting in a larger contact angle. Due to this, the metal may possibly no longer cover the entire bottom of the sapphire crucible, causing the light pulse to flash through the sample..

For measurement of metals with a very high surface tension, the new sample holder made of SiC is ideally



2 New sample holder for molten metals for the LFA 467 HT HyperFlash®.

suited (figure 2). In contrast with the conventional sample holder, this one has a thread screwing the SiC lid of the sample holder to the bottom, ensuring that the sapphire lid cannot move. It is thereby possible to prevent formation of droplets in the metal melt, thus achieving a defined thickness and entire wetting of the crucible bottom by the sample.



1 Left: Metal sample (solid) in a sapphire crucible with sapphire lid. Right: molten metal (beginning droplet formation) in a sapphire crucible with sapphire lid.

Material and Measurement Conditions

The material and measurement parameter employed are summarized in table 1.

Table 1 Material and measurement parameters

Instrument	LFA 467 HT HypeFlash®
Sample material	Copper, purity: 99.999%
Temperature range and sample holder	■ 25°C - 1200°C (new sapphire SiC sample holder)
	■ 225°C - 800°C (standard sample holder of alumina, round, 12.7 mm)

Results and Discussion

The new sample holder was put to the test by measuring a copper sample. The copper sample in the new sample holder was measured from 25°C to 1200°C into the melt. Melting of the sample can be recognized by the sharp decrease in thermal diffusivity (figure 3) and corresponds well with the literature value for the melting point (peak temperature) of copper, at $T=1083^{\circ}\text{C}$ [3]. For comparison, the copper sample was measured with a standard sample holder of alumina (12.7 mm, round) in the temperature range from 25°C to 800°C below the melt (gray diamonds in figure 3). The deviation between

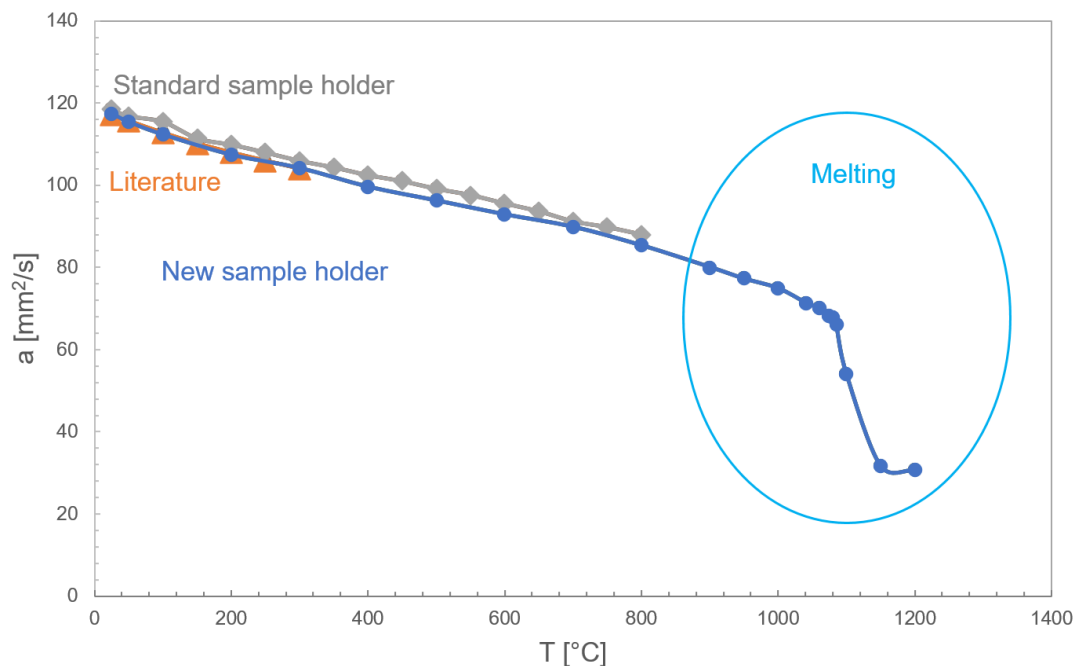
the thermal diffusivity values measured with the standard sample holder, the literature values (orange triangles in figure 3), and the values measured with the new sample holder for molten metals is less than 3% for all temperatures measured.

Summary

A new sample holder made of SiC for the temperature range from room temperature to 1250°C has been developed; it is ideal for the measurement of molten metals. The screwable lid ensures that the sample thickness does not change even in the melt, which is essential for accurate determination of the thermal diffusivity. Measurements on copper show good agreement of the results with those measured using a standard sample holder and also with literature values.

Literature

- [1] Dr. André Lindemann, Dr. Martin Brunner, LFA 467 HT Hyperflash®: Neuer Probenhalter – speziell für Metallschmelzen, AN 109, NETZSCH-Gerätebau GmbH
- [2] J. Schmitz, J. Brillo, I. Egry, *J Mater Sci*, 45, 2010, 2144-2149
- [3] J. A. Cahill, A.D. Kirschbaum, *J. Phys. Chem*, 66, 1962, 1080-1082
- [4] TPRC Database, 2005



3 Thermal diffusivity of copper (orange triangles: literature values [4], gray diamonds: measurement with a standard sample holder of alumina (12.7 mm, round), blue dots: measurement with the new sapphire Si sample holder for molten metals).