APPLICATION**NOTE**

Refractories – Laser Flash Analyse

Determination of the Thermal Conductivity of Refractories – The Solution: Investigation of Large Samples with the Laser Flash Analysis (LFA)

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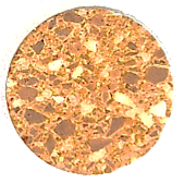

Introduction

Refractories are essential for high-temperature processes as they protect equipment used in steel, glass, ceramics, cement, chemical and energy engineering from extreme temperatures, aggressive substances and mechanical stress. They are used, for example, as linings in furnaces, reactors and melting tanks. A key material property in this context is the thermal conductivity. It significantly determines how much heat is transferred to the surroundings, directly influencing the energy efficiency of the process. Furthermore, the thermal conductivity has a significant effect on thermal stresses and consequently on the service life of the materials.

Refractories are inhomogeneous materials consisting of a matrix with embedded particles. When determining thermophysical properties such as the thermal conductivity, the following applies: The larger the sample, the more representative it is.

Determining the thermal conductivity of refractory materials presents a challenge to many measurement systems. This is due to two factors: the relatively high temperatures, typically exceeding 1000°C, and the inhomogeneity of the materials.

Method and Measurement Conditions

The LFA 707 *StratoFlash*® *Classic* can analyze samples with a diameter of up to 25.4 mm, even at high temperatures. The LFA method primarily determines the thermal diffusivity (α), and along with the density (ρ) and the specific heat capacity (c_p), the thermal conductivity (λ) is calculated using the following formula:

$$\lambda = \alpha \cdot c_p \cdot \rho$$

In the LFA method, the front surface of a sample is heated using a short energy pulse from a laser. The temperature increase on the back of the sample is then detected by an infrared (IR) detector. Mathematical models are then used to calculate the thermal conductivity based on this temperature increase.

The specific heat capacity can also be determined when the sample is analyzed alongside a reference sample. The most common method for determining the specific heat capacity at high temperatures is differential scanning calorimetry (DSC). However, typical sample sizes, with a diameter of 5 mm and a thickness of 1 mm, are not representative of refractories.

Using the large samples of the LFA 707 *StratoFlash*® *Classic*, with diameters of 25.4 mm, it is not only possible to determine the thermal diffusivity, but also the specific heat capacity on a representative sample using the comparative method in accordance with ASTM E 1461.

NGB · Application Note 426 · EN · 0326 · Technical specificationsa are subject to change.

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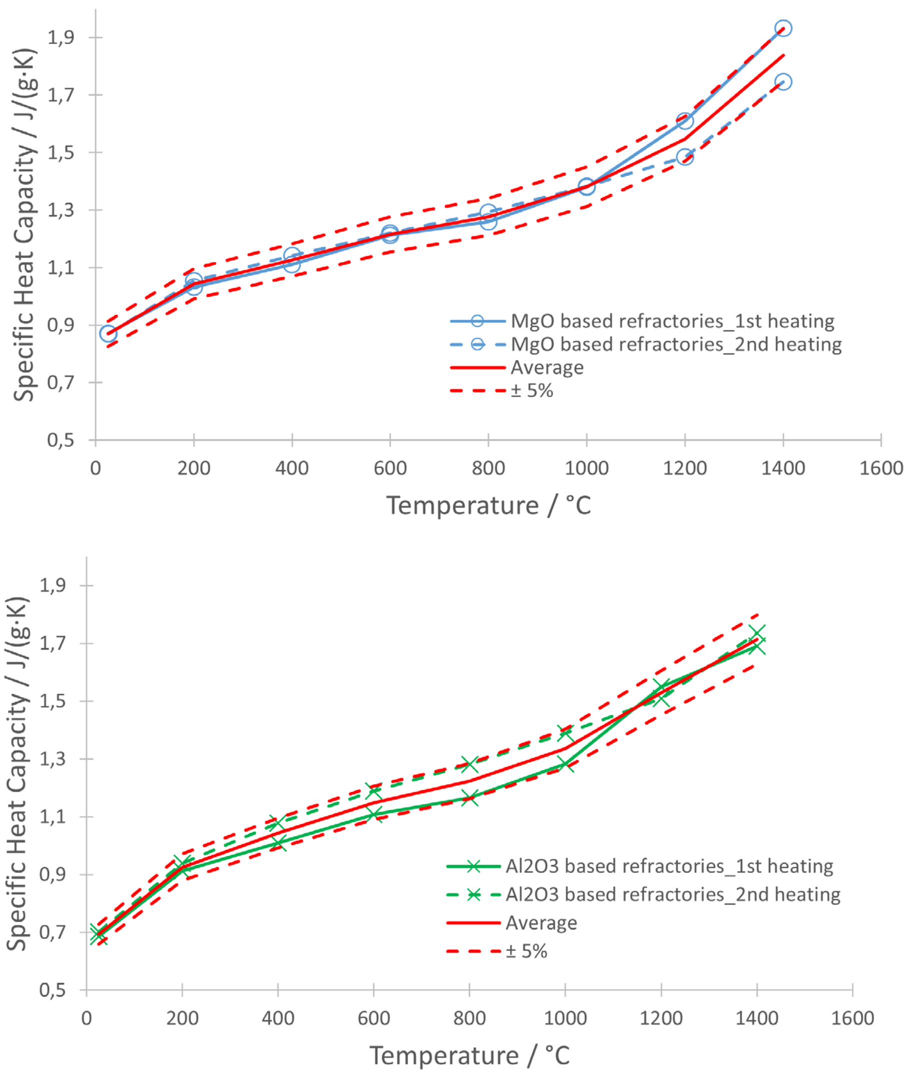
The measurement conditions are detailed in table 1.

Results and Discussion

Figure 1 shows the specific heat capacity of two refractory materials (MgO- and Al_2O_3-based) at temperatures ranging from room temperature to 1400°C. As expected, the specific heat capacity increases with rising temperatures. There is no significant difference apparent between the first and second heating cycles (within ±5%). This highlights the chemical stability of the sample (no decomposition and/or outgassing across the temperature range).

Table 1 Measurement conditions

Material	2 refractory materials on an MgO- and Al_2O_3- basis (thickness: approx. 3 mm)
Sample holder	Ø 25.4 mm, graphite
Temperature program	RT - 1400°C with 2 heatings
Sample size	Corresponding to material, one sample with Ø 25.4 mm and a thickness of ~3 mm, planparallel faces
Coating	Graphite
Reference for c_p	POCO graphite
Atmosphere	Ar
Heating rate	variable up to 20 K/min
Energy	600 V; 600 µs



1 Specific heat capacity of two refractories– two heatings each

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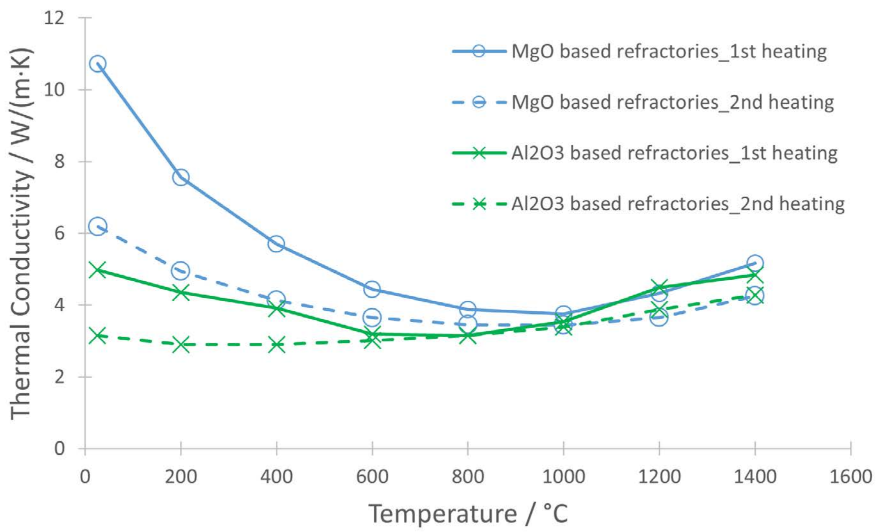

2 Thermal conductivity of two refractories – two heatings each

Figure 2 shows the thermal conductivity of the two materials, calculated using the aforementioned formula. In contrast to the specific heat capacity, clear differences are evident between the first and second heating cycles. These differences are likely due to structural changes within the sample (e.g., solid-solid phase transitions and/or formation of microcracks).

Summary

The LFA 707 *StratoFlash® Classic* is ideal for determining the thermal conductivity of inhomogeneous materials such as refractory materials due to its temperature range of up to 1600 °C and its capacity to accommodate large samples with a diameter of up to 25.4 mm. The device can also representatively determine the specific heat capacity. The resulting thermal conductivity is essential for designing and sizing equipment for high-temperature processes.

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