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Development and Setup of an Optical Dynamic Scanning Calorimetry Setup for Modern Material Science at High Temperatures

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Abstract

In modern high-temperature material science, currently a reliable measurement method for determining the specific heat capacity at temperatures far above 1000 °C is still lacking. In order to close this knowledge gap, a new measurement apparatus based on the differential scanning calorimetry method (DSC) is to be developed as part of the OptiMa project. Therein, radiation thermometry is used for the temperature measurement and various heating techniques will be examined.

Keywords: High-temperature applications, thermal analysis, differential scanning calorimetry, non-contact temperature measurement, radiation thermometry.

Motivation and Objectives of the Project

In many areas, modern materials science is of great importance for research and development projects. In additive manufacturing techniques, such as selective laser melting (SLM; cf. Fig. 1), material is melted locally at high temperatures using a laser beam. The goals of current development herein are to increase precision and improve process control. In order to increase the efficiency of thermal energy conversion processes, an increase in the process temperature is necessary, but this requires the development of high-temperature stable materials. Under operating conditions in gas turbines, the temperature of the ceramic thermal barrier coating (TBC; cf. Fig. 2) of the modern turbine blades is sometimes far above the melting point of the material of the metallic substrate.

The exact knowledge of the material parameters at high temperatures of 1000 °C to over 2000 °C is necessary for the reliable prediction of the behavior of various materials under the extreme operating conditions in the above-mentioned applications, for example. In particular, the thermal diffusivity and the specific heat capacity are of great relevance. To determine the thermal diffusivity, the laser flash method can be used as a verified measurement method even at high temperatures above 2000 °C. The specific heat capacity can already be measured reliably at temperatures below 1000 °C using the differential scanning calorimetry (DSC) method. However, for temperatures significantly above 1000 °C, there is currently no reliable measurement method available, that provides verified information about the specific heat capacity of material samples.

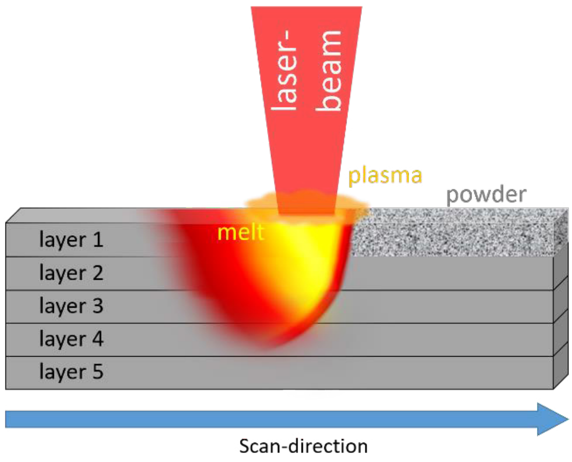

Fig. 1: Graphic illustration of the selective laser melting process (SLM) in additive manufacturing.

Therefore, the aim of the project *Optical Differential Scanning Calorimetry for Modern Material Research at High Temperatures (OptiMa)* is to remedy this lack of information in the future. As part of this project, the DSC method is to be further developed for use at temperatures between 1000 °C and over 2000 °C.

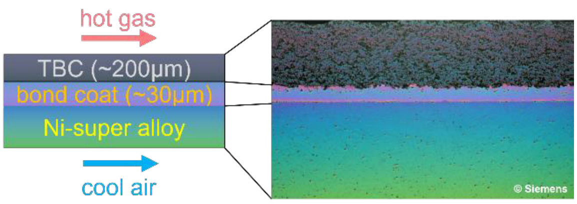

Fig. 2: Typical, of several layers consisting structure of thermal barrier coatings (TBC) on modern gas turbine blades.

Principle of differential scanning calorimetry

Differential scanning calorimetry is a method for determining the specific heat capacity. A typical measurement setup is shown schematically in Fig. 3. The material sample to be analyzed is inserted into a crucible, which is placed in the measuring cell together with an empty reference crucible. The entire measuring cell is heated with a specific temperature profile (e.g. continuous or stepwise increase). The temperatures of the heater (T_0), of the sample crucible (T_S) and of the reference crucible (T_R) are measured and the temperature difference (ΔT) between the crucibles is determined (cf. Fig. 4). A change in the course of the temperature difference indicates differences in the absorption of thermal energy and can be used to determine the specific heat capacity of the material sample under investigation after careful calibration of the setup [1][2][3].

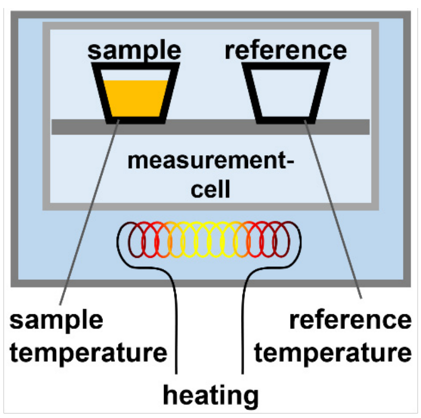

Fig. 3: Schematic drawing of a typical measuring cell for differential scanning calorimetry [1][2][3].

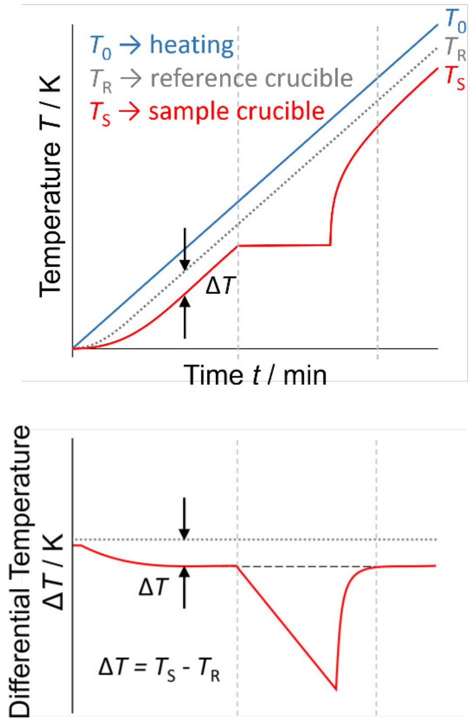

Fig. 4: Exemplary illustration of the course of the temperatures of the sample and reference crucible (top) and the differential temperature (bottom) during the heating process for a measurement using the differential scanning calorimetry method [1][2][3].

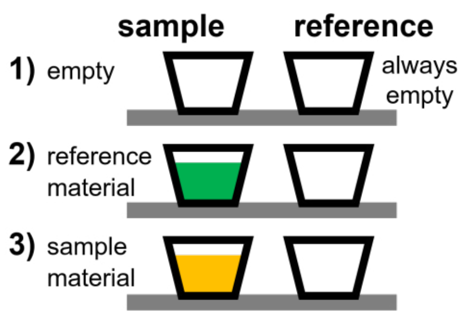

Fig. 5: Typical sequence of a measurement series using DSC:
1.) Empty measurement, both crucibles empty;
2.) Reference measurement, sample crucible: Reference material;
3.) Sample measurement, sample crucible: Sample material.

To calibrate the measurement cell (cf. Fig. 5), the measurement is carried out with two empty crucibles in a first step in order to obtain a baseline that takes into account the asymmetries of the setup. In the second step, the sample crucible is filled with a reference material of known specific heat capacity - e.g. sapphire. A calibration factor can be calculated from the result of this measurement, which can be used to determine the specific heat capacity of the sample under investigation [2][3][4].

Planned measurement method

In most DSC systems today, thermocouples are used for the necessary temperature measurement. However, the measurement uncertainty of these contact temperature sensors increases at higher temperatures. In addition, their service life decreases significantly. Therefore, today, reliable DSC measurements are only possible up to a maximum temperature of 1500 °C [5][6].

The measurement method planned as part of the OptiMa project (cf. Fig. 6) therefore intends to replace these thermocouples with methods based on contactless radiation thermometry. Specifically, a spatially resolved temperature measurement is to be carried out using high-resolution thermographic cameras. In addition, radiation thermometers are also to be used for temperature measurement. In particular, their measurement signal is to be used for heating control.

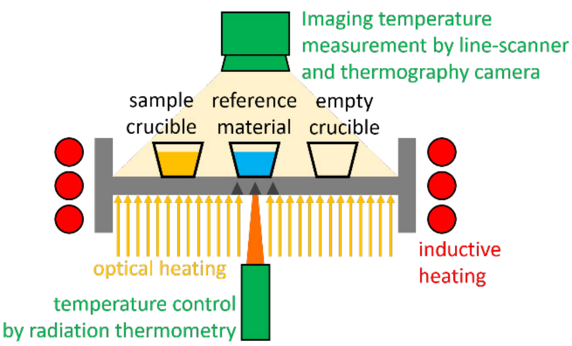

Fig. 6: Schematic drawing of the planned measurement setup for differential scanning calorimetry with temperature measurement by radiation thermometry and using a triple cell sample holder design as well as different heating techniques.

In addition to non-contact temperature measurement, several methods of sample heating are being investigated as a part of the project. Firstly, the measurement system consisting of the sample holder and the crucibles is heated inductively. For this purpose, the sample holder is made of electrically conductive and also high temperature stable material, e.g. graphite. In addition, the usability of an optical heating method for the sample holder system using laser radiation and a conventional electrical heating method using resistance-heating elements will also be investigated.

Furthermore, as part of the further developed DSC method, sample holders using a triple-cell design are to be applied. As a result, it will be possible to reduce the number of required single measurements. In addition to the sample measurement, only one reference measurement (all crucibles empty) is needed instead of two required reference measurements in a classic two-cell DSC system [7][8].

Experimental measurement setup

The central element of the new measuring set-up for optical differential scanning calorimetry (cf. Fig. 7) is a vacuum chamber mounted in a strut profile frame, which serves as a measuring cell and enables the heating of the samples to be examined in the absence of air. The surface of the measurement cell is temperature-stabilized in order to create a constant homogeneous radiation background to minimize the related measurement uncertainties. The measurement cell is equipped with several infrared-transparent windows. The measurement setup is also equipped with two movable traverses, which are used to mount measurement instruments such as thermographic cameras and radiation thermometers. Due to this combination, the samples to be examined can be observed from a variety of angles, and thus radiation thermometric measurement data can be recorded.

For heating the material samples to be examined, an induction heating system is used, which inductively heats the graphite sample holder following a predefined temperature course. In addition to inductive heating, sample heating using laser radiation is also being tested and evaluated as part of the project. The windows of the measuring cell therefore also allow the irradiation of a laser beam. In addition, the supporting strut profile frame is covered with opaque housing to ensure laser safety.

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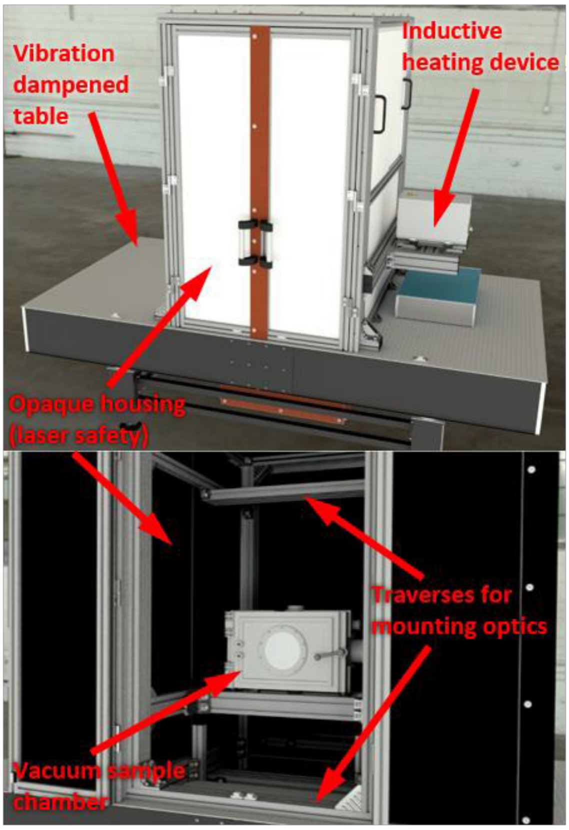

Fig. 7: Graphic depiction of the new measurement setup for optical differential scanning calorimetry.

Development of optimized sample holders

The sample holders used in the new DSC setup not only have to meet the requirements of the DSC measurement principle in terms of thermal conductivity, they also have to be selectively inductively heatable and also have to provide infrared-optical surface properties that are suitable for radiation thermometric temperature measurement. In order to meet all these requirements at the same time, specially adapted sample holders are also being developed for the new measurement setup. In order to optimize the sample holder geometry based on the triple-cell design according to the required properties, first detailed finite element simulations (cf. Fig. 8) are carried out so that design parameters can be varied without material loss. Using the knowledge gained from the simulations, sample holders are then manufactured for use in the first test measurements. In the course of this, the knowledge gained from the simulations can be verified in experiment.

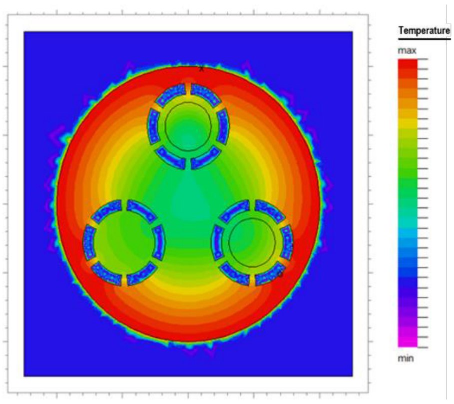

Fig. 8: Simulated temperature distribution on a variant of the investigated triple-cell sample holder design.

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