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Analysis Method

Temperature-dependent optical material properties of polymer powders regarding in-situ measurement techniques in additive manufacturing

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ABSTRACT

Due to the growing number of applications for Additive Manufacturing (AM), an increasing need for enhanced quality control methods exists. Here optical measurement techniques are often used for in-situ monitoring in AM. There is a great potential to standardize and improve applications of optical measurement devices in accordance to the increasing requirements of measuring tasks. Beside the optical material qualifications, especially process-oriented changes in optical effects are of great relevance for in-situ evaluations. The optical modifications may be attributed to aggregate specific phase transitions coming along with variable emitted wavelengths. A measuring setup with two integrations spheres and a heated process chamber have been developed to analyze the optical interactions of laser radiation and visual illumination with the powder. The process oriented analysis for selective laser sintering lead to improved operating conditions in the field of in-situ measurements in combination with an accurate evaluation of the absorption ratio of the examined powder.

1. Introduction

Additive manufacturing technologies allow significant time savings within the time-to-market process for developing new products [1,2]. By offering the possibility to flexibly build almost any geometry out of a CAD file, these technologies enable companies to achieve competitive advantages. For additive manufacturing selective laser sintered (SLS) polymer workpieces offer good mechanical part properties, like high tensile strengths [3] and are the choice for most industrial applications. Besides the advantages of SLS, different challenges needs to be met for successfully expanding the applications of the technology. One task is expanding the still limited material choice and the associated modification of new powder materials regarding their powder flowability [4], part porosity [5] or aging behavior [6]. Another task is the improvement of the quality control regarding the reproducibility of additive manufactured workpieces by different measurement systems. In this context, several in-situ measurement techniques are typically used for quality assurance [7]. There is a wide field of measuring applications from laser power measuring devices to mechanical measuring instruments as rotary encoders to determine the adjustment of the powder bed stage. But most established in-situ monitoring systems are based on optical imaging. Beside usual cameras, even high-speed cameras, thermography or photogrammetry systems are conventional components of an operating quality control system in AM. For using these systems the determination of the temperature-dependent optical properties of the powder material in solid and molten state is of importance. There are already different publications of other authors regarding the measurement of the optical material properties of polymer powders, which show different limitations [8–13]. In recent works *Laumer* presented an advancement of former measurement setups to determine the optical material characteristics of powder materials [14,15]. These setups lead to an advanced explanation model for describing optical material characteristics of polymer powders, which consider different level of absorptions by single powder particles in dependency on the laser wavelength [16]. In comparison to solid materials, there are multiple reflections in the powder bed leading to a high amount of laser radiation reflected back towards the powder surface. This radiation is partial absorbed by the particles and is thus attenuated. For in-situ measurements not the number of back reflected rays is determined, but the amount of energy of single rays, which sum up to the overall reflected radiation of the measured powder bed. A direct connection

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Table 1
Characteristics of the analyzed powders.

Material (Producer)	d10 in µm	d50 in µm	d90 in µm
Polyamide 12 (EOS)	32	55	74

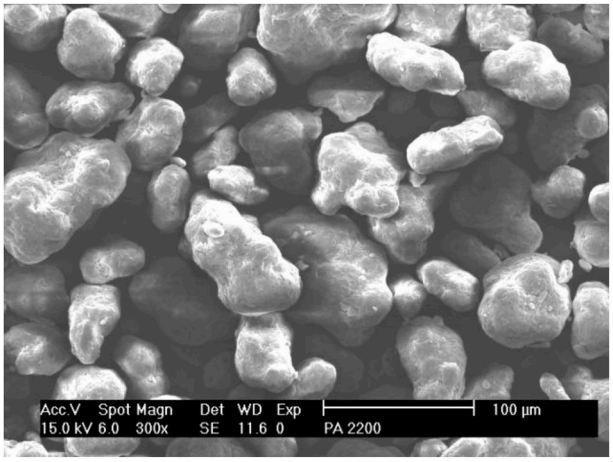

Fig. 1. SEM image of polyamide 12 powder.

between the absorptance of solid material and the overall optical material characteristics of powder material has been shown in Ref. [16]. However, the mentioned publications measure the optical material properties only at room temperature, although in SLS the material is heated up to its preheating temperature and finally molten by laser radiation. Especially phase transitions lead to a significant change of the optical material properties. Although there are first approaches to measure the temperature-dependent optical material properties of powder materials for other wavelengths [17], a detailed analysis of these properties for the SLS-relevant wavelength of a CO_2-laser is not yet available and represents the motivation of the following paper.

2. Experimental setup

2.1. Analyzed polymer powder

Each powder in AM has diverse material properties and thus behaves differently in dependency on the incoming energy input E_v. Consequently, a flexible measurement setup is needed to analyze different materials by providing various energy sources. Representative to other established powders in AM, polyamide 12 (PA12) is used for more than 90% of selective laser sintered workpieces [18] and analyzed within this research. Beside the following evaluations of this examined PA2200 powder from EOS concerning the optical properties in dependency of the transition phase, also other materials may be characterized within the developed measurement setup in future works. In contrast to investigations of reflectivity effects with solid workpieces, also the absorption behavior during the additive manufacturing process is of great relevance. A relevant condition for the evaluation of the absorption ratio is a transparent or at least semi-transparent layer to determine the transmittance of the material by a measurement device. In this context the thickness of the powder layer, its optical characteristics, the particle geometry and even the particle size distribution of the powder are relevant. Consequently pre-investigations are necessary to analyze these influencing factors before measuring the optical properties of the powder. Thus, the particle size of the PA12 powder has been examined as seen in Table 1. The particle size distribution indicates that 50% of all examined particles are smaller than 55 µm. In a further research the particle geometry has been analyzed by a scanning electron microscope (SEM). This SEM image PA12 in Fig. 1 illustrates the detected powder distribution.

2.2. Measurement setup

The measurement setup consists of two integration spheres, as shown in Fig. 2. They allow a measurement of the temperature-dependent reflectance ratio R, the transmittance T and consequently the absorptance A. A collimated laser beam is guided into the reflection sphere and passes the thin powder layer on the specimen holder between the integration spheres. In the following described experiments a

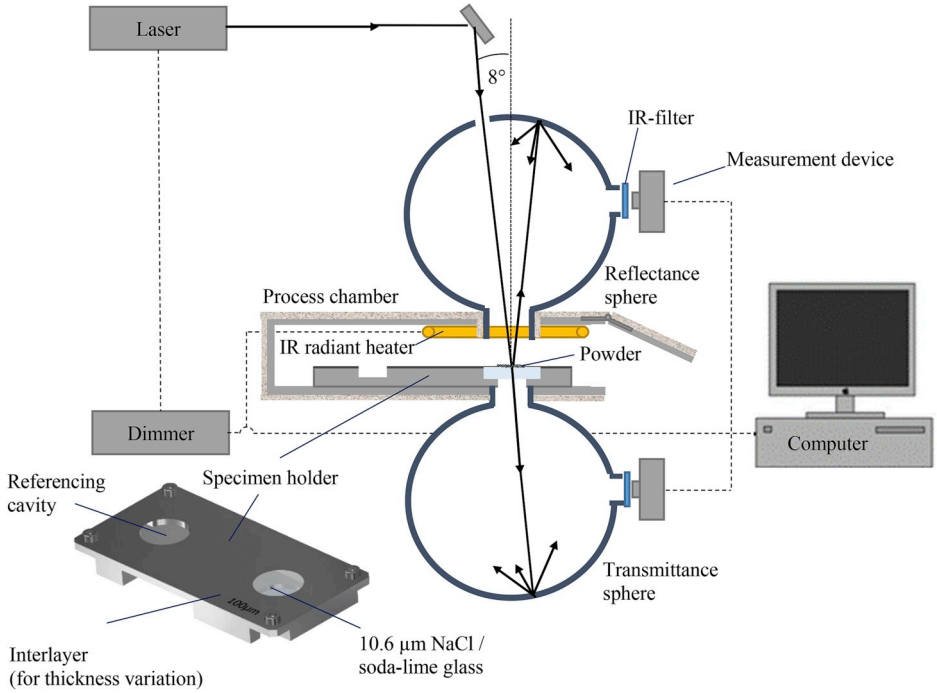

Fig. 2. Integration spheres measurement setup.