



On the selection and design of powder materials for laser additive manufacturing



Lasers used in production processes such as additive manufacturing are becoming more powerful and flexible, but the materials available often have limitations for today's laser processing tasks. Hence, more focused research is needed on materials design and preparation (typically in powder form) for laser additive manufacturing to shape the process chain right from the start.

This Preface places the focus on increasing the library of materials employed in laser-based additive manufacturing, enhancing or providing new functionalities to the existing ones, and adapting/optimizing their processing. The relevance of this topic is evident since only few metal and polymer powder materials available for laser-based additive manufacturing fulfill the requirements to comply with up-to-date process routes. Many new powder materials show insufficient characteristics regarding their processability and resulting part properties. To address those limitations, advancements on this field require the expertise and cooperation between researchers from materials science as well as photonics, showing how these two fields can work together to produce next generation metal, polymer and composite powders.

As an example of the current materials limitations in additive manufacturing (AM), it should be noted that metal powders are still employed that were developed more than 50 years ago for a completely different kind of process, i.e. thermal spraying. In modern laser-based additive methods, however, these powders result in undesired part properties such as residual porosities and other defects in the component. Even when optimized process conditions are used, batch-to-batch variations of chemical composition, particle size distribution or particle morphology lead to process instabilities. Furthermore, available metal alloy powders are often optimized for conventional processing routes (e. g. casting, milling, etc.) without due regard for the unique characteristics of laser-based processes (e.g. high solidification rates). There are already examples of powder materials or alloy compositions that have been successfully adapted to photonic processing proving a clear enhancement of the generated part properties [1–4]. In the field of polymer powders for laser powder bed fusion (LPBF) there is also a lack of variety of raw materials. Polyamide (PA) 12 is used in more than 90% of the LPBF built polymer parts, while other material types such as high-performance, e.g. PEEK, thermoplastic elastomers, TPE, and ultrahigh-molecular-weight PE is increasing [5]. Typical problems of new polymer powder materials are their insufficient flowability [6] and high losses of the laser power due to high reflectance of the powder materials [7]. Hence, there is need to adapt these metal and polymer powder materials to the popular production processes, since laser-assisted techniques are set to gain importance in production processes

in the long term due to their high throughput as well as their great precision [8–10].

A very successful example of material adaption to processing and production to enable them to perform new functions can be found in the field of mobile devices: in more than half of all smartphones, laser direct structuring is used nowadays to write complex circuit layouts of copper directly on the plastic platform. This method was made possible by adapting the function of the plastic material used – via use of an additive – which is activated by a laser beam, leading to reductive copper plating by means of a physico-chemical reaction [11]. Another example of a successful material development is “Scalmalloy”, a high-strength aluminum alloy powder designed to be processed by laser-based AM. This hypereutectic aluminum alloy strengthens the aluminum matrix by the precipitation of nanometer-scaled particles, leading to superior mechanical properties [12]. Due to the high cooling rates and rapid solidification during AM, unique microstructures can be achieved to allow the material to rival the performance of aluminum foundry products. This alloy cannot be used in casting due to the low cooling rates involved, that lead to coarse precipitates with little effect on the mechanical properties. These two examples indicate that the material development methodology for specific laser-based AM needs has a considerable but largely untapped potential.

In laser-based additive manufacturing (Fig. 1a), powder materials are predominantly used (Fig. 1b). When all the critical success factors of additive manufacturing processes are considered, the (powder) material emerges as one of the most important [12], followed by the machine control, throughput and productivity. Fluctuations in the powder quality in terms of the morphology and grain size as well as the composition can presently not be compensated by process engineering measures, or only to a limited extent. However, certain methods drawn from materials science and chemistry enable the modification of powder materials, e.g., by the incorporation of (nano)additives to adapt the powder properties to the properties of the laser radiation and to modify the material evolution during processing (Fig. 2a–b). This allows altering such properties of the final material as the hardness or tensile strength, or providing new functionalities. To achieve this, different strategies have been shown to be successful for metal and polymer powder processing. The contributions within the Virtual Special Issue reflect a significant part of current research trends in the field, focusing on the following routes/goals:

- Material development for increasing the powder and built part quality and increasing the selection range of AM-processable materials [15–18].

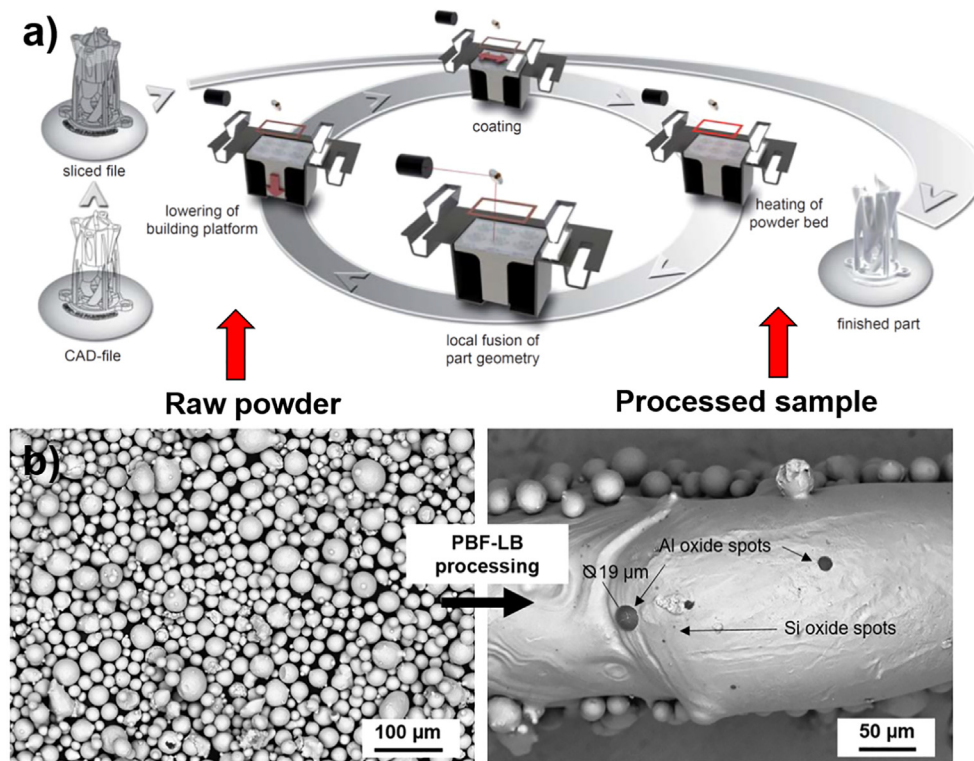


Fig. 1. a) Schematic illustration of the cyclic layer-by-layer generation process during laser powder bed fusion (LPBF). Reproduced with permission from ref. [13]. b) Illustrative SEM images of the initial powder material employed in additive manufacturing and the resulting structure after processing. Reproduced with permission from ref. [14].

- Research on utilizing (nano-)additives incorporated within or deposited on the powder materials to influence the laser process [19–22].
- Macroscopic and mesoscopic simulations to support the development of new materials [23,24].
- Physico-chemical analysis of the material-process correlation enabling predictive material development [25–27].

Even though highly advanced additive manufacturing systems are available, with different laser sources (e.g. diode lasers), the current knowledge base of the complex interaction mechanism and different influencing factors regarding the material properties and the laser processing parameters needs to be further expanded. Up to now, the research in the field of laser beam interaction focuses on the understanding of the interaction of laser radiation with already existing powder materials, such as PA12 [30,31] or 316L stainless steel [32–34]. Consequently, an in-depth evaluation of the processes that take place during laser-based additive manufacturing (Fig. 2c) is necessary to widen the library of available materials. However, there exist large differences between metal and polymer powders as well as between processing techniques and parameters. Hence, the specific challenges in metal and polymer powders for laser-based AM are described in the following sections to detail and to highlight the advances proposed by the contributions to the Virtual Special Issue and in the related literature.

1. Metal powders

Metallic powders have a great application spectrum and enormous potential for additive laser processes [35]. Several metallic materials used for AM include steel and iron-based alloys, titanium and titanium-based alloys, nickel-based alloys, aluminum alloys, cobalt-based alloys, copper alloys and precious metals [36–39]. While there are numerous available base materials, compared to conventional processing routes, the library is still very limited. Metal powders should

generally be weldable to be successfully processed by AM avoiding process-related cracking. Apart from that, other parameters that affect AM fabrication are processability (flowability, wettability), and optical (absorption), mechanical (strength) and functional properties (conductivity, magnetic response).

The parameters listed above are determined by the raw powder properties and the (nano)additives, if present. In that sense, the synthesis methodology is required to ensure the reproducibility of the powder properties. Techniques like gas or water atomization either by crucible-based or inductive heating still lead to difficulties in producing materials of sufficiently high quality, especially for alloy systems exhibiting high sensitivity to contamination by impurities, such as oxygen, or moisture [40] (e.g. titanium alloys). As an example, the porosity of AlSi10Mg volume bodies produced by LPBF is decisively influenced by the moisture content of the powder material leading to the formation of pores. Since porosities and component defects cannot be completely avoided during processing of conventional metal powders, when metallic powders are used for additive manufacture, often a thermal posttreatment is necessary to reduce the porosity and residual stress [41,42].

The synthesis process exerts a significant influence on the morphology and thermomechanical properties of the generated powders. However, it is not the only factor that can influence the base powder properties. The incorporation of nano-additives has been shown to be a successful strategy to modify or provide new functionalities to the powders [43–47]. In that sense, A. Lüddecke et al. [19] studied the influence of SiC, a few atomic layers of graphene and iron oxide black on the absorption and wettability of stainless steel, tool steel and aluminum alloy. The results show that the (nano)additivation improves the absorption at the processing laser wavelength as well as the flowability of the powders. The work by O. Pannitz et al. [22] with a stainless steel powder enriched with a few layers of graphene and SiC reports improvement in the thermal conductivity of the powder, leading to faster heat dissipation and higher temperatures during processing (Fig. 3a). The studies of aluminum oxide, nitride and titanium carbide indicate

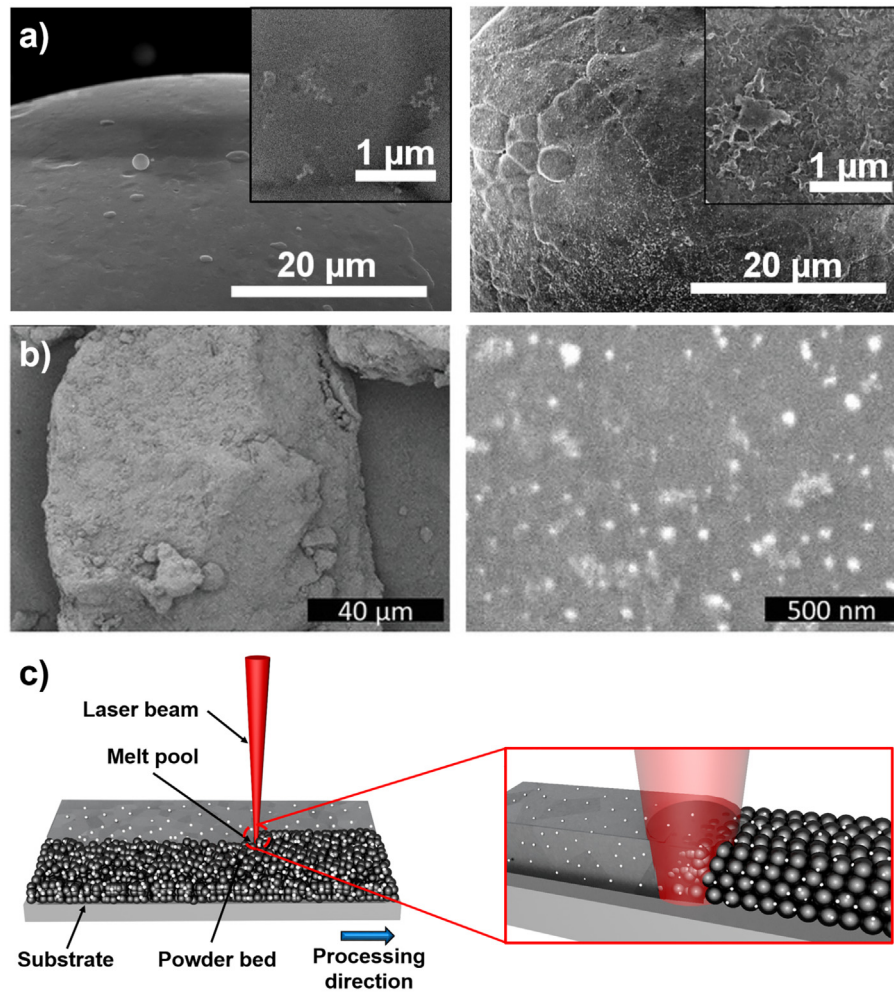


Fig. 2. Combination of micro-powders with nanoparticles for additive manufacturing: (a) Scanning electron micrographs of gas atomized tool steel powder (left) and steel powder decorated with yttrium oxide nanoparticles (right). Reproduced with permission from ref. [28]. (b) Scanning electron micrographs of TPU powder decorated with silver nanoparticles. Reproduced with permission from ref. [29]. (c) Schematic diagrams of the processing of these microparticles by laser powder bed fusion.

[48] that the wetting properties exert a considerable influence on the powder processability and the resulting hardness improvement of the tool steel contour produced. This effect can be observed for magnesium alloys, which are highly interesting for fabricating lightweight parts or individual biodegradable implants. Due to the high reactivity and the rapid formation of oxide layers, high-energy input is required to break the oxide and to achieve wetting of the structure by the metallic melt. It has been shown that the processability of magnesium powder materials can be significantly increased by the addition of a more reactive alloying element such as yttrium and neodymium, which reduces magnesium oxide formation and improves mechanical performance [49]. In the case of a CuCr powder the employment of the carburized alloy and optimization of the thermal post treatment leads to a 18% increase in conductivity [21]. The addition of oxide nanoparticles, e.g. Y_2O_3 was shown to improve the mechanical properties of fabricated parts through the formation of oxide dispersion strengthened steels (ODS). This material type has attracted high interest due to its enhanced mechanical properties at high temperatures and radiation resistance (Fig. 3b) [50,51].

Another effect that can occur during laser processing of alloy particles is an undesirable change in the alloy compositions due to different vapor pressures, solution properties and reactivities of the alloying elements. For the Cu-Al system, different values of copper content in aluminum wrought alloys were achieved by decomposing copper acetate in a reactor and simultaneously depositing copper onto aluminum

particles to thermally process them with laser radiation [52]. The eutectic Al-Ni also shows compositional variations depending on the processing technique, with the temperature-dependent concentration interval within which the two-phase eutectic coupled growth takes place shifting towards higher concentrations when the solidification velocity increases [24].

AM has the potential to create high performance alloys with unique microstructures [4,53]. The particular feature of laser-based additive manufacturing concerns the very high cooling rates in the melt pool [54]. Rapid solidification leads to changes in the resulting microstructures compared to casting techniques, characterized by tremendous grain refining. High solidification rates also enable a significant increase of the available alloy spectrum, e.g. immiscible or supersaturated alloy compositions (e.g. Cu-W) [55]. By this method, the beneficial properties of specific elements (e.g. high electric conductivity of copper and high melting point of tungsten) can be combined in a single material for improved macroscopic physical and mechanical properties [56].

The *in situ* synthesis of reinforced metallic materials with ceramic particles by laser-based processes has been demonstrated for different material systems, proving the ability to reduce flotation tendencies by rapid solidification and strong Marangoni convection forces within the melt pool [51,57,58]. Some alloys designed for other processing routes, e.g. forming, are also processable via AM [59]. However, many alloys that are of interest for additive manufacturing are not processable, such as high strength wrought aluminum alloys. An example is EN

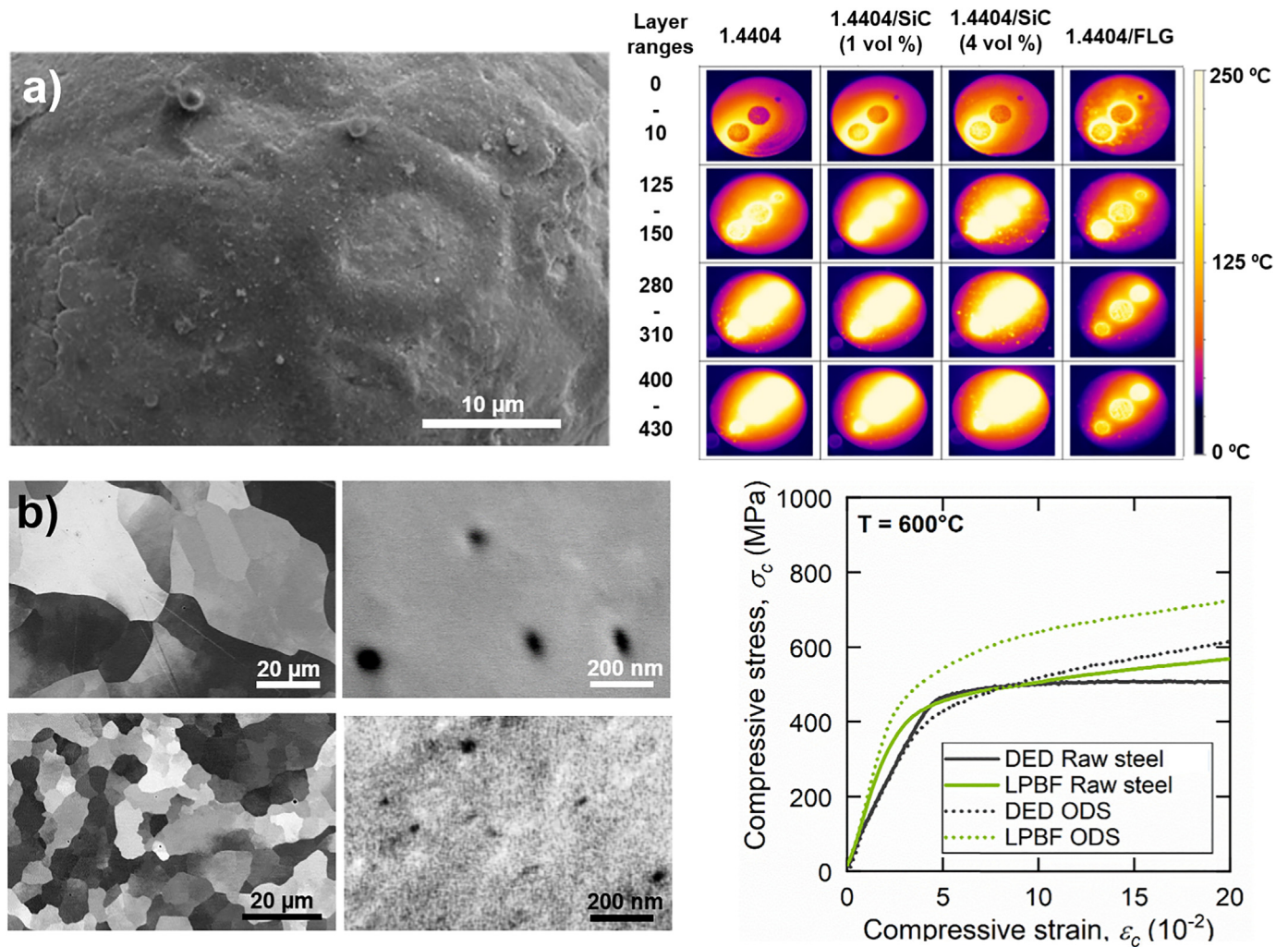


Fig. 3. a) SEM image of stainless steel nanoadditivated with 4 vol% of SiC (left). Temperature distribution in the powder bed of stainless steel 1.4404 and coatings influence (right). Adapted with permission from ref. [22]. b) SEM images of 0.08 wt% Y₂O₃ nanoadditivated iron chromium powder processed by LPBF (top-left) and directed energy deposition (bottom-left). Corresponding compressive stress measurements at 600 °C (right). Adapted with permission from ref. [50].

AW7075, exhibits high hot cracking susceptibility during AM, typically leading to widespread crack networks across manufactured parts. Sistiaga et al. demonstrated significantly reduction of the cracking susceptibility of an aluminum wrought alloy powder by adding 4 wt% Si particles which leads to defect-free parts [60]. Similarly, Martin et al. showed that small amounts of ZrH₂ nanoparticles can induce heterogeneous nucleation and facilitate equiaxed grain growth, thereby reducing the effect of solidification strain and preventing hot cracking [1]. In another study, Haase et al. demonstrated superior mechanical properties of an additively manufactured high-entropy alloy compared to conventional casting [61].

Adapting alloy systems for laser-based AM processes also allows the utilization of the rather complex thermal history of laser-produced parts, where the material experiences cyclic reheating. This can also be used for the design of new materials as shown by Raabe and co-workers who exploited this “intrinsic heat treatment” to induce the precipitation of NiAl nanoparticles in a Fe-19Ni-xAl model maraging steel, hereby strengthening the laser-produced part [62]. Using this characteristic feature permits the omission of a post-build heat treatment, which is commonly necessary for additive manufactured parts [63]. Prashanth et al. also demonstrated the modification of mechanical properties of Al-12Si by adapting scan strategies in the LPBF process proving the enormous potential of simultaneous process- and material adaptations [64].

From the state-of-the-art in the field it can be seen that the powder material properties have a considerable influence on the laser printing processability. However, it is also clear that there is a need for interdisciplinary approaches to adapt to the meltability or flowability of metal powders, but also to the wettability, as well as the optical properties. The results arising from the current Virtual Special Issue reveal the potential of different additives (SiC, graphene, iron oxide black and amorphous C) to adapt the optical properties and wettability of the metal powders. In fact, these additives were shown to influence the functional properties of the printed objects such as electrical conductivity (C) and thermal conductivity (graphene and SiC), as well as mechanical properties (Y₂O₃).

2. Polymer powders

Polymers offer excellent possibilities of “custom-made” manufacturing, due to the diversity in their molecular structure, and the cost-effective material base and production processes. However, restrictions arise from the limited temperature stability and the mechanical properties of the final component. High potential is attributed to lab-on-a-chip applications [65], microsystem technology and smart systems in micro- and nanoelectronics [66], as well as in biomedicine [67]. Further economic potential is seen in the electronics, automotive, aerospace, and machine applications [68].

The main method of laser-based additive manufacturing using polymer powders is LPBF. While initially polymer components made by LPBF were only used as models or as prototypes, today LPBF of polymers is of great interest for the "custom-made" production of technically sophisticated components, especially for the automotive and electronics industries. However, the currently available range of basic materials, let alone their modifications, is still limited. Even though some materials are emerging, it represents a significant obstacle to the further development of the research field [5,69]. More than 90% of commercial powder materials for laser powder bed fusion consist of polyamide 12 and polyamide 11. A few forms of PEEK and thermoplastic elastomers (TPU, TPE) are also commercially available for laser-based manufacturing [70]. The transition from "rapid prototyping" to "additive manufacturing" has also led to an increase in the demand for manufactured parts in recent years. Today, however, the same material properties as (for instance) obtained from injection molding cannot be achieved in any way other than by additive manufacturing [71]. The restriction in the choice of material is based on the requirements of the sintering process [72]. Thus, only semi-crystalline polymers with the thermal softening and cooling behavior best suited for the process can be used in a narrow processing window [30]. For TPU, for example, these requirements lead to a very small process window. Problems of component stability are also caused by the additive process itself, which leads to numerous material

interfaces, and thus to phase boundaries with changes in crystallinity and adhesion in the component. High demands arise for the rheology and optical properties of the powders as a result of the process that limit considerably the type of additives and fillers that can be used. A significant expansion of the material variety for LPBF, therefore, requires more understanding of the melting and crystallization behavior and its interplay with additives and fillers in thermoplastics. Still relatively unexplored for LPBF are highly interesting biodegradable polymers such as PLA [73] and technical thermoplastics from chain polymerization reactions such as ABS and TPU. Also, the possibility of a better control of the particle shape and size distribution, possible by polymerization processes (dispersion polymerization, (mini) emulsion) are still under development [74]. The expansion of the material variety and the determination of optimal thermophysical powder properties can be supported by process simulations that predict the temperatures, temperature gradients, heating and cooling rates and mechanical stresses occurring in the process [23]. It is thus clear that numerous new polymer powder materials (PLA, TPU, potentially ABS) as well as formulations and processing strategies (emulsions, additives, co-precipitation) ought to be investigated further in relation to the process behavior during additive manufacturing.

In the field of plastic powders, there are exemplary works by Schmidt et al. [18,75] for optimized powder production with regard to

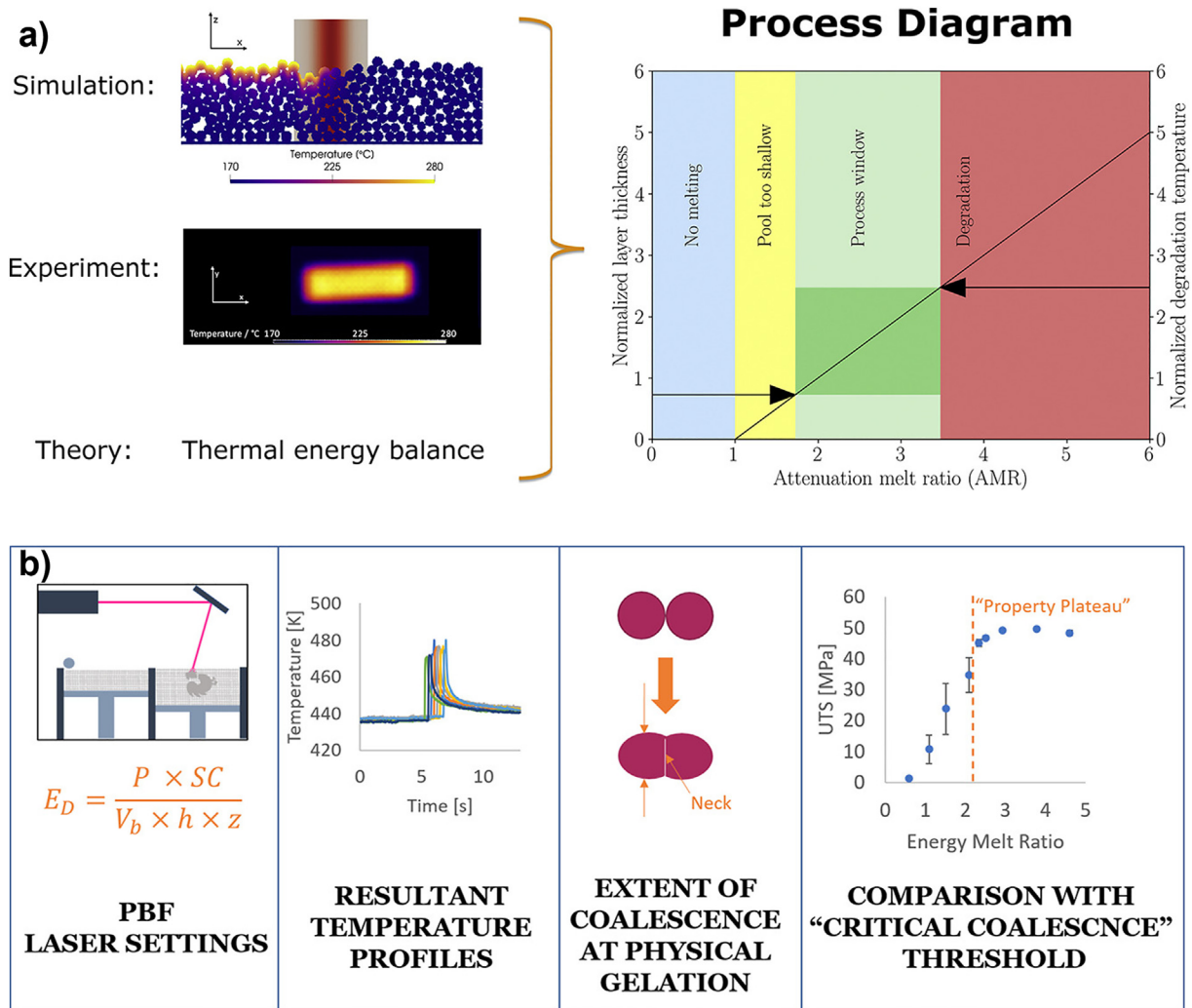


Fig. 4. a) Scheme of the proposed methodology to calculate the processing window of polymer powders processed by LPBF from its thermomechanical and optical properties. Reproduced with permission from ref. [23]. b) Proposed model based on the particle coalescence to relate the parameters employed in polymer LPBF with the mechanical properties of the generated parts. Reproduced with permission from ref. [25].

the particle size and flowability. The importance of size distribution, morphology, thermal, rheological and optical properties of the polymer particles for laser powder bed fusion is also evaluated. To increase the flowability and improve the process behavior of plastic powders, the following approaches can be found in the literature: rounding in the fall tower [76], modification of surface chemistry / additive [77] and particle production from melt emulsions [78]. The influence of the flowability and the shape of the powder particles on the powder application process were numerically investigated in [79]. Using a discrete element simulation, the properties of a powder coating applied in the process were modeled and analyzed as a function of the powder properties.

Modeling and simulation can advance the development of new powder materials for additive manufacturing processes in numerous ways. It is possible to analyze numerically the characteristics of powders which do not yet exist and cannot be tested experimentally. In the simulations it is also possible to determine process variables that are impossible or hard to be measured in the experiment, such as heating or cooling rates. On the macroscopic scale, i.e. considering the complete process, thermal and thermomechanical simulations can predict temperature fields, temperature gradients, heating and cooling rates, and residual stresses. The sensitivities of these field variables with respect to the material parameters can be identified and used to formulate recommendations for *material design*, a topic of central significance for the journal titled *Materials & Design*. On the mesoscopic scale, i.e. the length scale where the particular powder particles are explicitly modeled, sub-processes such as powder application, the energy input or melting and consolidation can be simulated. The influence of the flowability and the shape of the powder particles on the powder application process were numerically investigated in [79]. Melting of the powder particles and consolidation were addressed by a Lattice Boltzmann method e.g. in [80,81] to study the melt pool characteristics and

the re-solidification of the molten material. These mesoscopic simulations allow studying the influence on the powder processability of various thermophysical properties, e.g. the absorption coefficient. Furthermore, a method of theoretical calculation of the attenuation melt ratio (AMR) and the processing window of polymer powders is proposed by Bierwisch et al. and confirmed with the experimental data from PA12 and PEEK [23]. This approach together with the numerical, analytical and experimental investigations to identify a dimensionless number to characterize the LPBF process of PA12 [26] represents an advancement towards modelling the LPBF process to obtain the processing window and the required parameters for any polymer materials from their base thermomechanical and optical properties (Fig. 4a). In fact, Chatham et al. [25] propose a model based on the critical coalescence ratio that relates the heat transfer and temperature profiles obtained from the thermal and optical properties of a Nylon 12 powder with the tensile strength and density of the generated part (Fig. 4b).

As described for the metal powders, the addition of nanoparticles within or on the surface of polymer powders also leads to significant changes in their laser processability. Due to its broad absorption spectrum, carbon black is the most widely used nanoscale additive. Typical methods to incorporate (nano)additives include direct mixing procedures such as milling, as well as melt compounding [82], solution intercalation [83], in situ polymerization [74], ex situ dispersion into polymer solution [84] and adsorption of nanoparticles in aqueous solution [85,86]. The method employed has a decisive influence on the degree of dispersion, mechanical properties and morphology of the powders. A typical challenge during mixing of nanoparticles by the most often used method, i.e., milling, is agglomeration. A high degree of agglomeration, in turn, leads to the need for higher degrees of filling in order to increase the surface coverage. It is, therefore, essential for LPBF that the dispersion of the nanoparticles within the polymer matrix (Fig. 5) and the interfacial bonding between filler particles and filler-matrix

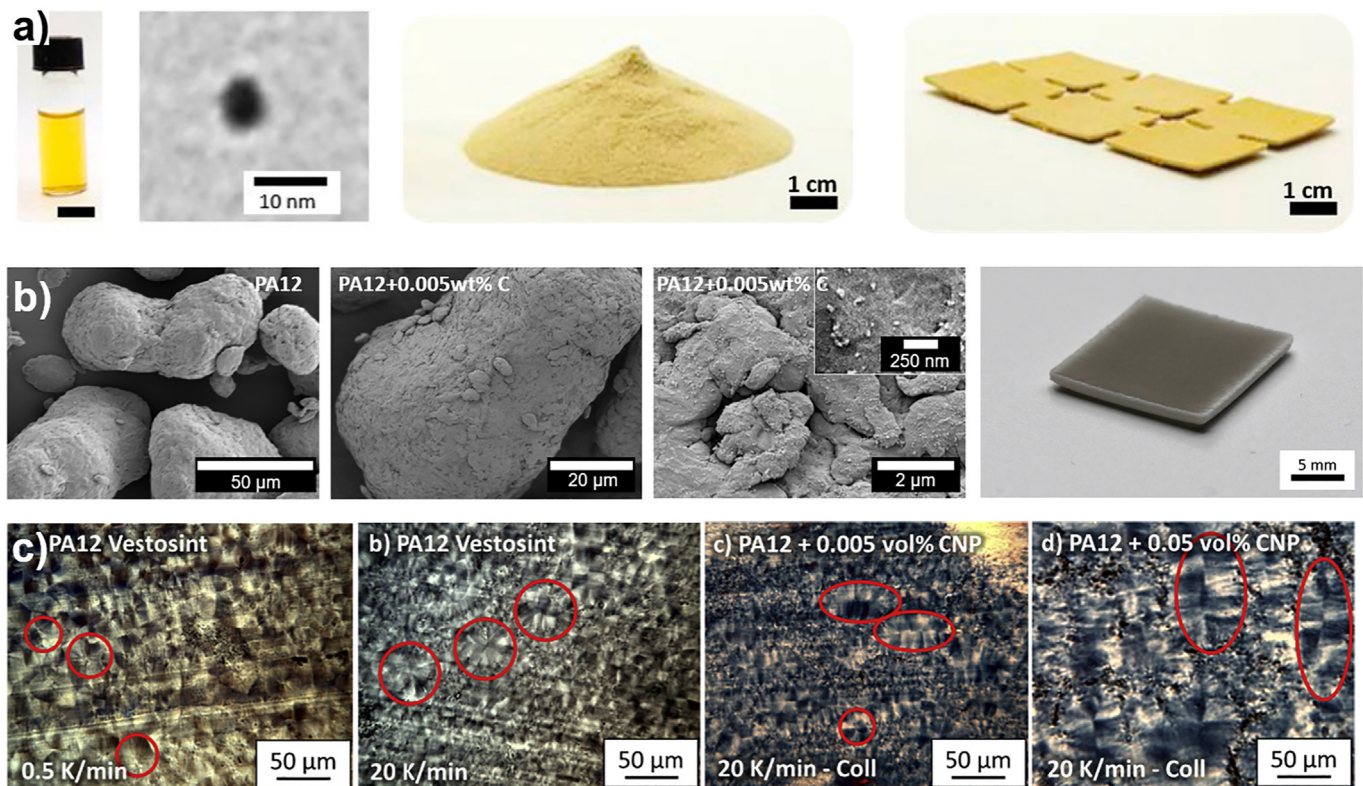


Fig. 5. a) Additive of PA12 powder with plasmonic Ag nanoparticles and printed part by diode laser LPBF. Reproduced with permission from ref. [87]. b) Carbon black supporting on PA12 achieving a homogeneous dispersion. Reproduced with permission from ref. [88]. c) Evaluation of the carbon black vol% effect over the microstructure of LPBF printed PA12 and PA12-carbon black powders. Reproduced with permission from ref. [20].

[31] are optimized while the mass fraction of additives is minimized. Also, LPBF process parameters may remain unchanged if the filling factor is low, as shown for <0.1wt% of carbon black by Kim et al. [31] and Sommereyns et al. [20].

The reliability and reproducibility of production quality represents a further important challenge for LPBF, that is closely related to material properties. There is a lack of quality and standardization concepts considering the number and variety of the influencing factors along the entire process chain. Schmid and co-workers presented a study in which these factors were analyzed to obtain a reliable material data set for different testing parameters [89]. They also proposed characterization methods that do not depend on the powder batch and the amount of used powder, but are based on solution viscosity, molecular weight distribution, differential scanning calorimetry (DSC) and particles size distribution. In another study, Pham et al. used gel permeation chromatography to compare the molar weight of the polymer powder before and after sintering and revealed that significant ageing (i.e. change of crystallinity and melting point) occurs, further indicating the need for quality control [90]. These limited studies show that quality and standardization concepts are crucial for ensuring reproducible results in laser-based AM.

For both metal and polymer powders there are current limitations for laser-based additive manufacturing that can only be overcome by targeted material synthesis and adaptation to the process. In addition, requirements and deficiencies are often found to be applicable to both material classes. These challenges need to be addressed by interdisciplinary collaborative research that involves considering the numerous existing material-based ideas and approaches. The examples presented in this Preface show that the preparation of the starting material is a key factor and that there is a need to drive further research to modify or develop materials for laser-based methods to optimize laser processability and establish the additive process route for new material classes. Furthermore, specifically tailored polymer materials and metallic alloys are needed to unleash the full potential of additive manufacturing technologies and increasing the benefits for industrial application. The contributions of the *Materials & Design* Virtual Special Issue *Materials for Laser Additive Manufacturing* represent a step forward in that direction, providing a clear framework from the theoretical and experimental point of view for the future development of novel materials for laser-based AM.

Declaration of Competing Interest

None.

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