Additive Manufacturing of Gradient and Multimaterial Components

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Abstract
In the paper first results regarding the realisation of gradient and multi-material parts manufactured by Laser Beam Melting in powder bed of metals and polymers are published. Gradient properties of additively manufactured metal parts can be achieved by varying the composition of alloying components in the powder and adapting process strategies. As an alternative to atomizing pre-alloyed materials, mixtures of different powders are investigated. For realizing multi-material-parts from polymers, at first relevant material properties concerning compatibility have to be analysed. Therefore the paper shows the main requirements for compatibility between different materials and also first results regarding the compatibility of polymer powders and possible combinations for the manufacturing of multi-material components by laser beam melting of polymers.

Keywords
Laser Beam Melting in Powder Bed, New Materials, Additive Manufacturing

1 INTRODUCTION

Laser Beam Melting (LBM) is a manufacturing technique which is nowadays mainly used to build prototypes from a single material. Melting powder layer by layer offers geometrical freedom that cannot be achieved otherwise. This potential for complex-shaped work pieces is rarely exploited so far. The use of multi-material and gradient materials enables the combination of different materials and the variation of their microstructure in one part. Such composite structures can fulfil different product requirements within a single building process, whereas at the moment such devices are assembled from several independently produced parts. Saving process steps enables lower costs compared to the conventional production.

In order to reach this aim further research in polymeric and metallic Laser Beam Melting is necessary.

2 GRADIENT COMPONENTS BY LASER MELTING OF METALS

2.1 Motivation

Processing aluminium alloys by means of LBM in powder bed is considered well understood [1, 2]. Alloys close to the eutectic AlSi12 (e.g. AlSi10Mg) are by far the most commonly used ones. These alloys have proven to be well suited for the LBM process [3]. For processing with manufacturing methods other than LBM, a large range of alloys is available, which offer superior mechanical properties due to hardening mechanisms, often triggered by certain heat treatments. The choice already offered today by LBM to either melt any given volume element or have it remaining as loose metal powder shall be transferred to material properties and scaled down to the level of microstructure. Powder systems with particles of different sizes and elemental composition are being developed for that purpose. Opposed to state-of-the-art pre-alloyed metal powders, these allow different properties resulting from metallurgical microstructures realized by locally adjusted laser processing conditions in addition to flexible adaptation of alloy compositions. Powders of different materials with different densities tend to separate from each other after being mixed. A way to reduce this de-mixing effect is the preparation of nano-particle coatings on micro-particles [4, 5, 6].

In this contribution, the suitability of such coated powders for the process of Laser Beam Melting is investigated on the examples of the aluminium alloys EN AW 6061 and 2024.

2.2 Experimental

2.2.1 Powder coating with nano-particles

As a host material for the powder mixtures aluminium powder with a purity of 99,8 weight percent is used. Utilising a vibration sieve shaker Haver & Boecker EML 200 digital plus, powder particles bigger than 40 µm and smaller than 20 µm are removed.

Alloying elements are added in the form of particles with diameters between 25 and 60 nm, except for Magnesium, which is not available as a metallic nanoparticle and hence added as powder with particle sizes between 20 and 63 µm. The elemental composition of the two powder coated alloys covered in this paper can be seen in Table 1.

The powders are weighed on a precision scale and put into gas tight cylindrical mixing containers of 200 mm height and 100 mm diameter. Sieving, weighing and packaging are performed in a Glovebox providing an atmosphere with less than 1 ppm of oxygen content in order to prevent the nano-particles from oxidation. The mixing containers are shaken in a Turbula T2F mixer by manufacturer Bachofen for 90 minutes at 40 rotations per minute.

	Alloying components [weight-%]						
EN AW	**Al**	**Cu**	**Si**	**Mg**	**Mn**	**Ti**	**Fe**
2024	94,4	4		1	0,6		
6061	97,6	0,4	0,6	1,2	0,15	0,15	0,5
Size in µm	20 < Al < 40			20 < Mg < 63			
Size in nm		40	50		40	60	25

Table 1 - Alloying components of EN AW 2024 and 6061

As results of this mixing procedure, in scanning electron pictures the nano-particle coatings can be seen, which is shown in Figure 1 and 2. The bigger powder particles are covered with smaller nano-particles. The nano-particles tend to agglomerate. During mixing in the Turbula Shaker, agglomerates are broken due to impact forces when crashing with the micro-particles. Some agglomerates of different sizes stick as a whole to the micro-particles, as can be seen in Figure 2. The distribution of nanoparticles on the surface of the bigger particles in Figure 1 appears to be more homogeneous. Because of the small sample shown in the pictures compared to the total amount of mixed powder, SEM pictures can only give hints, but hardly provide a quantitative analysis of the mixture quality.

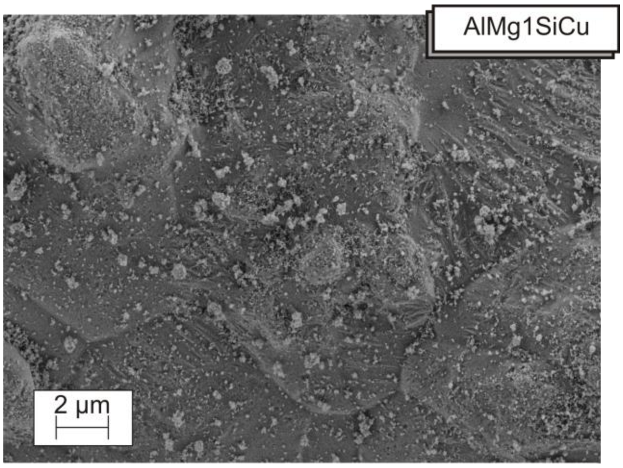

Figure 1 - SEM-picture of coated EN AW 6061

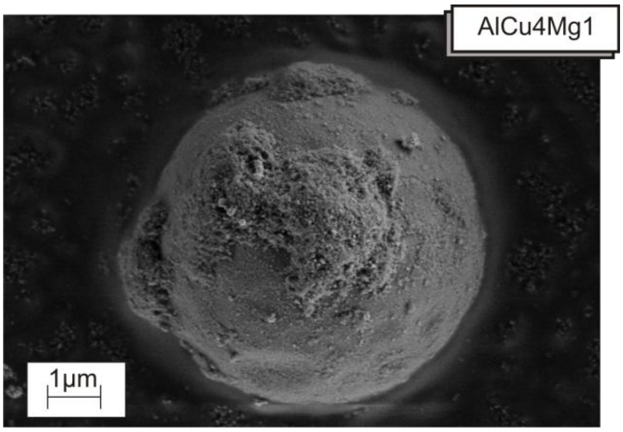

Figure 2 – SEM picture of coated EN AW 2024

2.2.2 Tensile Testing of powders

Coated nano-particles act similar to surface roughness on the larger particles. They minimize potential contact surfaces and serve as spacers, reducing inter-particular adhesion forces which decrease with distance. This effect can be determined with a powder-tensile-test according to Schweiger and Anstett [5]. Figure 3 illustrates the setup which is used in lateral view (on the left) and in front viw (on the right). A powder sample is compressed under a cylinder, and subsequently the force required to lift it off is measured. In order to make the powder stick to the cylinder, it is sprayed before with 5 % petroleum jelly solved in petroleum ether.

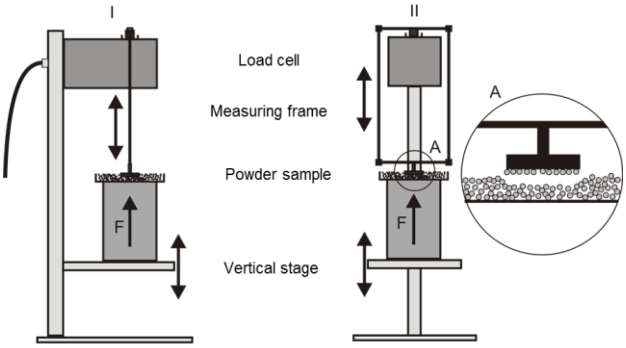

Figure 3 - Powder tensile test setup

Results of tensile tests with standard pre-alloyed aluminium EN AW 2024 powder are shown in Figure 5 in comparison to Figure 4 showing EN AW 2024 realised as a coating of nano-particles on 99,8 % pure aluminium host particles. Each test has been performed 7 times. Depending on pre-compression, the coated powder exhibits a tension which is more than 20 times lower than the pre-alloyed powder. Particles of the pre-alloyed powder are in the same range of size as the host particles for the coated powder. Additionally, the shape of the pre-alloyed particles is more spherical compared to the pure aluminium particles, which are more irregular in size, reminiscent of potatoes. Such irregularities in shape can increase tensile test values due to interlocking.

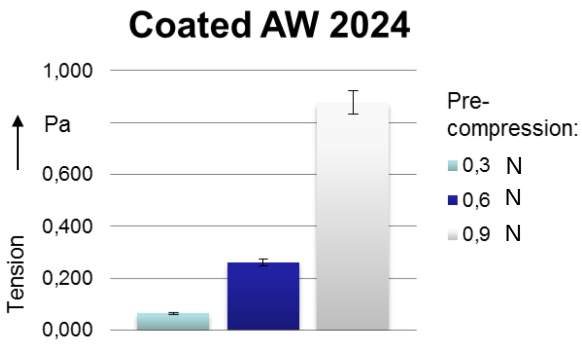

Figure 4 - Tensions after pre-compressions with nano-particles

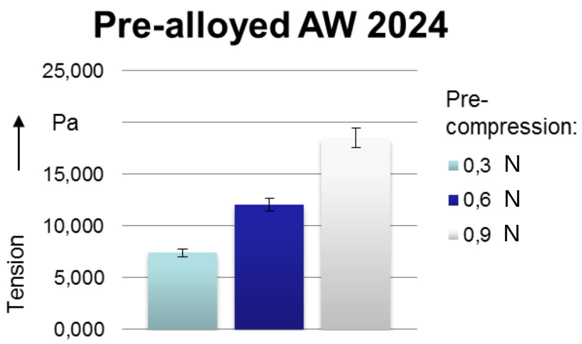

Figure 5 - Tensions after pre-compressions without nano-particles

2.2.3 LBM-processing of nano-coated powders

For laser beam melting experiments, a commercially available machine is used: SLM 50 by manufacturer ReaLizer. Test specimens are built in the shape of cubes with 5 mm edge length. As could be expected from the tensile test results, layer coating in the SLM 50 machine is much more even and homogeneous with the coated powders than with pre-alloyed. Yet, no quantitative analysis of the layer quality has been conducted.

A ground micro-section of a laser beam molten cube is shown in Figure 6. The low visible porosity exemplifies the processability of this coated powder mixture.

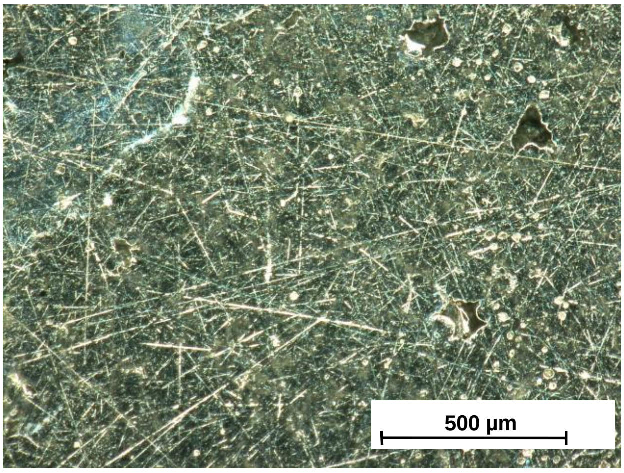

Figure 6 – Micro-section of Al 6061 after LBM

3 COMPATIBILITY OF POLYMER POWDERS FOR MULTI-MATERIAL COMPONENTS

3.1 Theoretical Background

For the realisation of multi-material components the identification of compatible polymer powders is essential. Otherwise the forming of a connection between different materials in the laser melting process is not possible.

There are several requirements for compatibility between two different materials which altogether affect the compatibility.

One important requirement is the compatibility regarding the process temperatures of the used materials, further called temperature compatibility. For the laser melting process the material must have a common melting range. Otherwise one material is pyrolysed before the other material reaches its melting point. This compatibility requirement can be analysed by differential scanning calorimetry (DSC) [7].

Another requirement is the chemical and physical compatibility. Hereunder material characteristics like surface tension, polarity, wetting behaviour and the molecular weight of the materials are of importance. This information is strongly dependent on the polymer and has to be analysed for each powder material separately. Especially for material characteristics like surface tension or wetting behaviour there are no common analysis methods for polymer powders available yet.

Another important factor influencing the compatibility of different materials is the process and temperature controlling during the manufacturing process. There is a direct relation between the formation of adhesion mechanism and heat transfer processes. Also the geometry of the boundary layer is of great importance for the forming of cohesion between different materials.

3.2 Experimental setup for the analysis of the compatibility

3.2.1 Used powders and analysis of the powders involved

One problem is the limited choice of materials in the field of polymer laser melting. The different powders which can be bought and were used are PA 2200 (PA 12; EOS), a polyamide 12 based powder, polypropylene (PP; DuPont), polyethylene (PE-HD; DuPont). Other materials have to be milled in order to get powder out of injection moulding granulate. In cooperation with a local firm small amounts of polyoxymethylene (POM), polyethylene terephthalate (PET), polylactic acid (PLA) and polybutylene terephtalate (PBT) were produced in order to analyse the compatibility in a broader material range.

All powders were sieved with a 120 µm sieve to make sure that the maximal particle size is below this limitation. In order to know the melting range of the different thermoplastic powders DSC analyses were carried out. Therefore small amounts of powders were heated and cooled down in order to discern the melting and crystallisation point. By comparing the different melting ranges it is possible to determine combinations which cannot be used because of non-overlapping melting ranges.

3.2.2 Analysis of compatibility

The most important experiment is a melting test involving the different materials. Therefore two different powders were allocated besides each other in one setting and on top of each other in a second setting. The powder is set in two metal rings like illustrated in Figure 7. The inner ring had a diameter of 10 mm, the outer diameter 20 mm. One powder was always coloured with 0.1 weight percent carbon black in order to identify the different materials. This small amount of additive doesn't affect the material characteristics [8]. The powder setting was then put in a preheated oven for 5 min whereby the temperature of the oven was within the overlapping melting range of the polymer powders. Afterwards the oven was chilled down to room temperature, before the probes were removed.

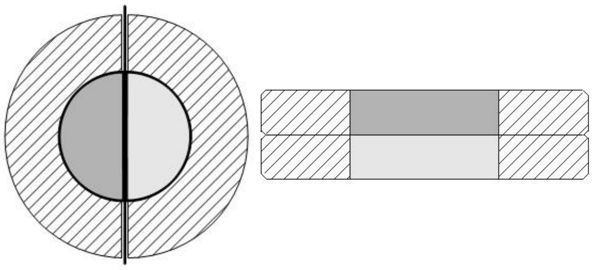

Figure 7 - Experimental setup

After the removal from the metal rings cut images were made in order to obtain a qualitative result of the experiment.

3.3 Results and Discussion

3.3.1 Results of the powder analysis

The powder analysis by DSC shows the significant processing temperatures like the melting and crystallisation point. And thus gives basic data to compare the melting ranges of different materials. The melting range of all used powder materials can be seen in Table 2.

Material	T_M [°C]	T_C [°C]	ΔT [°C]
PA 12	184	141	43
PP	155	122	33
PE-HD	132	117	15
POM	163	145	18
PET	253	192	61
PLA	160	139	21
PBT	212	198	14

Table 2 - DSC analysis results of the different materials

Most of the materials are temperature compatible with some exceptions. For example, the melting point of PET with 253° C is so high that PA 12, PP and PE powders are already pyrolising before PET starts to melt. Therefore this combination is not compatible.

3.3.2 Results of the compatibility analysis

The melting experiments are a easy method to prove the compatibility of different powders in a qualitative way. Figure 8 shows a picture of carbon black coloured PE-HD and white PP.

The combination can easily be removed from the metal rings and indicates a good connection between the two materials. The overall results of the melting experiments are presented in Figure 9.

Figure 8 - Picture of a cut sample with PE-HD powder (on top) and PP powder

Figure 9 shows several combination possibilities. The “P” marked combinations means that one material was pyrolysed because the melting ranges of the powders were different.

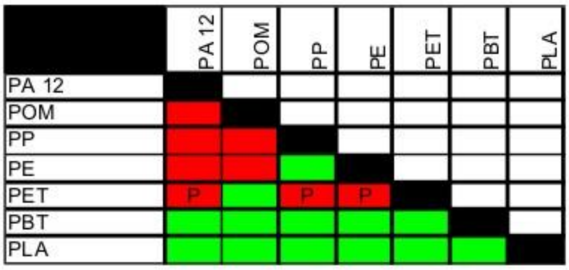

Figure 9 - Compatibility matrix

4 CONCLUSIONS

4.1 Conclusions Metal

Powder alloys have been created by coating micro-particles with nano-scaled metallic particles, which has been verified by SEM pictures. Apart from stabilizing mixtures, interparticular attraction forces are reduced compared to pre-alloyed standard powders. On the example of coated EN AW 6061, processability to almost dense parts on an unmodified commercial LBM machine is demonstrated.

4.2 Outlook Metal

In future works, additional alloys are to be composed of pure powder mixtures. Optimal parameters for the mixing procedure are to be determined and de-mixing phenomena of nano-particle coatings to be quantified. Locally different microstructures shall be created from those mixtures using different optimised process parameters. Chemical and mechanical analysis of LBM-processed samples are to be performed.

4.3 Conclusion Polymer

The most important requirements for the compatibility of different powder materials were shown. In first experiments several compatible material combinations have been found which offer new possibilities for multi-material components manufactured by selective laser melting of polymers.

4.4 Outlook Polymer

In future works, test methods are researched for achieving results for tensile strength or other important characteristics to obtain also quantitative information about the adhesion strength between different powder materials.

Furthermore an adequate multi-material deposition mechanism and an adjusted process controlling is needed in order to realise multi-material components.

5 ACKNOWLEDGEMENTS

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7 BIOGRAPHY

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