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Investigation of aging processes of Ti-6Al-4V powder material in laser melting

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Abstract

Laser melting of titanium material, e.g. Ti-6Al-4V, offers great potential in manufacturing automotive components, lightweight structures and medical implants. In order to achieve required mechanical properties of laser melted components quality of powder materials is essential. Unmelted powder is recycled and reused in a subsequent process. Due to repeated recycling it is suggested that powder material changes. In this paper aging processes of Ti-6Al-4V powder are studied. It was observed that powder particles coarsen and flowability increases. Comparing examined powder characteristics to bulk material properties it was noticed that there are significant effects of aged powder on laser melted components.

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Keywords: laser melting; Ti-6Al-4V powder; particle size distribution; bulk material properties

1. Introduction

In recent years, components made of titanium and its alloys, e.g. Ti-6Al-4V, became increasingly important, especially in automotive and aircraft industry as well as in the field of medical technology. Titanium materials can be characterized by a high specific strength, an excellent corrosion resistance and a good biocompatibility. However, raw materials production and materials processing involve high costs [1]. In order to optimize resource management and manufacturing costs an increased rate of materials utilization is desirable from an environmental as well as from an economic point of view.

The technology of laser melting for manufacturing near-net-shape components fosters savings of natural resources and costs along the value chain. In comparison to conventional production processes

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(casting or milling) laser melting enables automated manufacturing without any tools and offers great potential for production of highly individual and complex parts with almost unlimited geometrical freedom in design.

In laser melting a laser beam is used to selectively melt thin layers (20 μm – 50 μm) of metallic material in a powder bed. Powder material is distributed across a building platform using a recoater device. Interaction between powder particles and laser beam causes the material to melt. After solidification of the exposed area a new powder layer is applied. That way three-dimensional parts are created corresponding to a 3D-CAD model in a cyclic process. Unmelted metal powder is sieved and reused in a subsequent process.

Functional parts produced by laser melting only rank among the state-of-the-art in niche markets such as dental industry and tool making [2, 3]. Currently, there are efforts to establish laser melting of Ti-6Al-4V in series production for different applications [4]. In order to successfully implement this technology in industry quality demands have to be fulfilled. Quality of laser melted parts depends amongst other things on density, hardness, dimensional accuracy and mechanical properties.

An essential prerequisite to achieve Ti-6Al-4V components with optimal properties are purity and adequate condition of powder material used in the process. Due to the large surface of metallic powders there is a risk of chemical reaction between Ti-6Al-4V powder material and oxygen, carbon, nitrogen and hydrogen of the ambient atmosphere [5, 6]. These gases attach to the surface of the particles and form oxide and nitride layers. Hence, bond forces between powder particles as well as powder flowability are influenced. Additionally, it was observed that powder condition, especially flowability of steel material, changes due to repeated recycling [7]. It was also noticed that the particle size distribution which is significant for powder bed density has an effect on density, mechanical properties and surface roughness of manufactured components from stainless steel powders [8, 9, 10].

The present paper focuses on a systematic study of aging processes of Ti-6Al-4V powder material which has been reused several times for laser melting. Powder characteristics such as particle size distribution and flowability are examined over a specific period and related to laser melted bulk material properties.

2. Experimental

The material used for laser melting of test specimens was a commercially available gas atomized Ti-6Al-4V powder with a particle size D90 of approximately 50 μm according to the materials certificate. All test specimens were produced using an industrial state-of-the-art system equipped with a 200 W fiber laser, standard exposure parameters and a layer thickness of 30 μm . Post processing of test specimens was not carried out.

2.1. Powder analysis

Ti-6Al-4V powder material was used in laser melting processes over a period of several months for 12 cycles. After each process excessive powder was sieved and reintroduced into a subsequent production cycle. Powder preparation by sieving was carried out within the system under ambient conditions. For classification purposes a sieve with a mesh size of 80 μm was used. Virgin Ti-6Al-4V material was characterized upon receipt. In addition, Ti-6Al-4V powder material was analyzed previous to every manufacturing process regarding particle size distribution and particle shape using laser diffraction as well as a Scanning Electron Microscope (SEM). Furthermore, a qualitative evaluation of powder flowability by means of visual analysis and the determination of apparent density were carried out.

2.2. Determination of bulk material properties

In order to evaluate the quality of manufactured parts and relate it to determined powder characteristics several test cubes and tensile specimens were laser melted during each production cycle. Test cubes were analyzed with respect to density, porosity, hardness and surface quality. Density was measured according to Archimedes method [11]. Porosity was examined with the help of light microscopy. Hardness of test cubes was determined following Vickers hardness testing. Surface roughness was studied using laser confocal microscopy. Moreover, mechanical properties of test specimens were determined by tensile test.

3. Results and discussion

3.1. Results of powder analysis

Virgin Ti-6Al-4V material was characterized upon receipt. Fig 1 shows the particle size distribution and particle shape of the material.

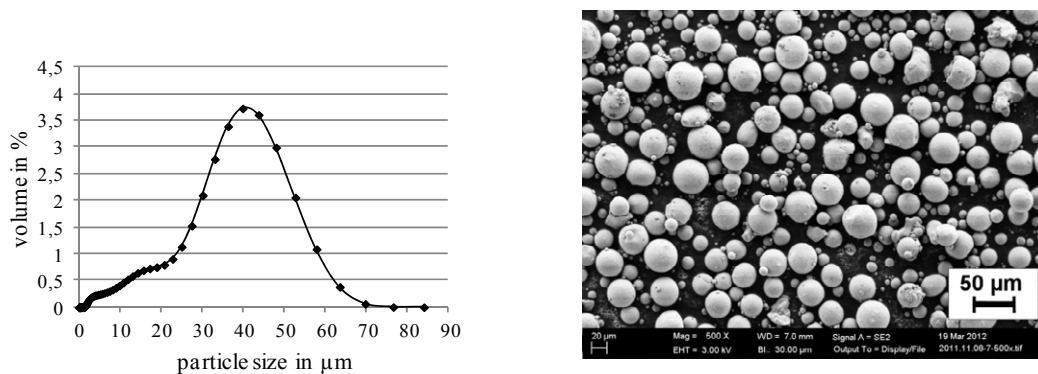


Fig. 1. (a) particle size distribution of virgin Ti-6Al-4V; (b) particle shape of virgin Ti-6Al-4V

The distribution of powder particles is Gaussian-like but with a slightly higher proportion of fine particles. The average particle size amounts to 37.4 μm and maximum particle size is about 70 μm . The majority of particles is spherical.

Measured particle size distribution of powder after several production cycles is shown in Fig 2. It can be clearly recognized that particle size distribution changes due to multiple recycling. The average particle size increases up to 51.18 μm after 12 cycles. Less particles with a size < 20 μm are detected. The proportion of powder particles with the highest volume is reduced and the size distribution is enlarged. Particles with a size > 100 μm could be determined.

It was observed that particle size increases as a consequence of sintering processes because of residual heat close to the melt pool and due to adhered Ti-6Al-4V particles which come off as soon as the component is removed from powder bed (cf. Fig 2 (b)). Small powder particles with a size of less than 20 μm are raised inside the process chamber during sieving of excessive material after the process. These particles remain in the ambient atmosphere and are distributed. Thus, fine powder particles are not recycled and reintroduced in a subsequent production cycle.

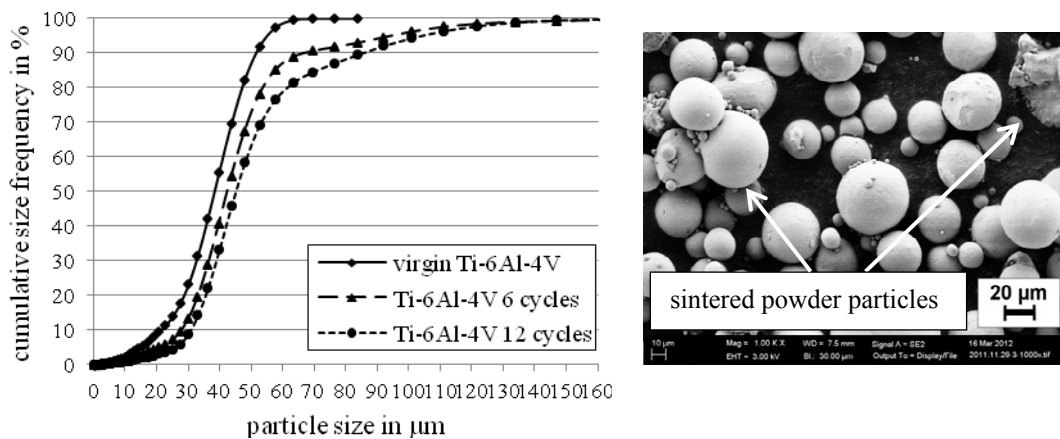


Fig. 2. (a) comparison of particle size distribution; (b) sintered powder particles

Virgin Ti-6Al-4V material has an apparent density of 2.27 g/cm^3 and a comparatively poor flowability. This is suggested by a comparison of Fig. 3 (a) and (b) which show the results of the qualitative flowability analysis. The high proportion of fine particles gives rise to agglomeration effects. Agglomeration affects flowability of powder. Flowability as well as apparent density of the powder increase as the aging of the material proceeds. After 12 production cycles apparent density amounts to 2.47 g/cm^3 . This can be explained with a coarsening of powder and a simultaneous reduction of adhesion and friction between single particles.

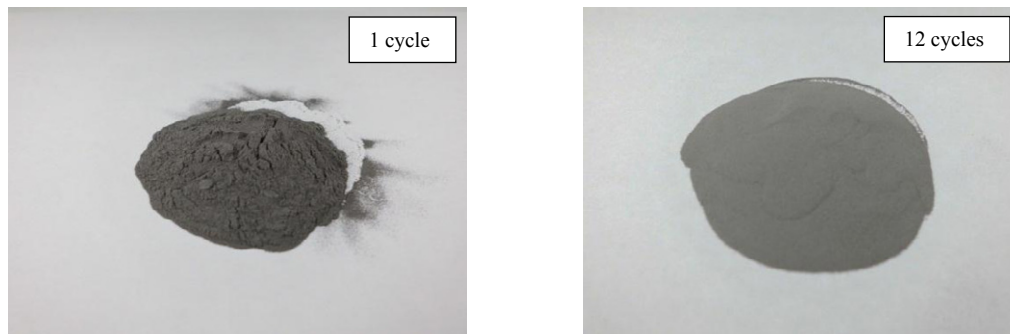


Fig. 3. Flowability of (a) virgin Ti-6Al-4V; (b) reused Ti-6Al-4V (12 production cycles)

3.2. Properties of laser melted test specimens

Density of test cubes laser melted using virgin Ti-6Al-4V is at 4.408 g/cm^3 and lower than density of test cubes manufactured using recycled powder material (cf. Fig. 4 (a)). This result can be related to the poorer flowability of virgin powder material. As the material agglomerates particle distribution on the building platform is influenced.

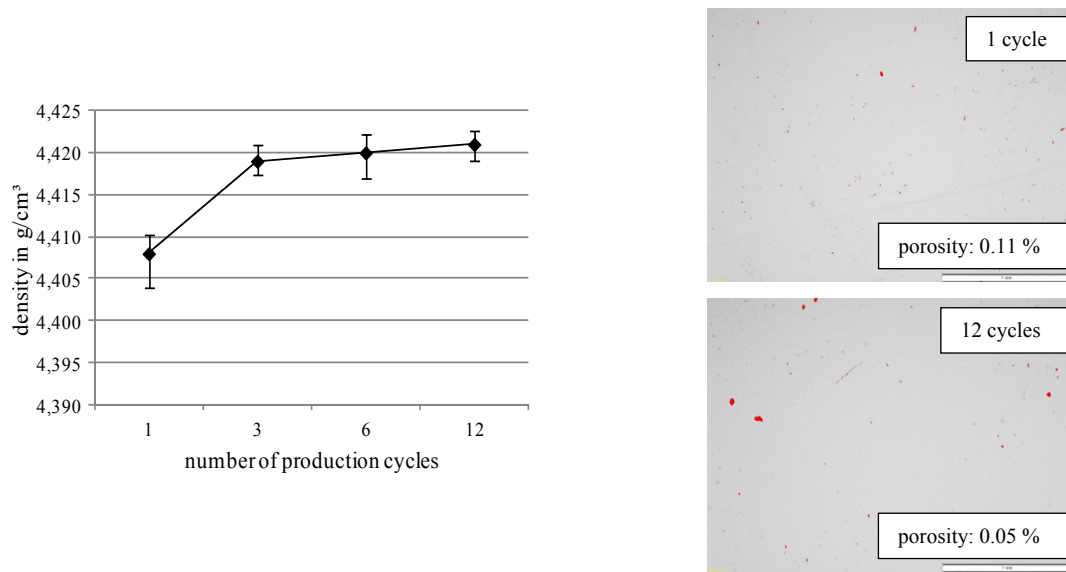


Fig. 4. (a) density of laser melted bulk material; (b) porosity of virgin Ti-6Al-4V and reused Ti-6Al-4V (12 production cycles)

With an increasing number of production cycles density of laser melted test specimens increases up to 4.421 g/cm³. This is due to a wider interval of particle size distribution which favors higher density because of better particle packing characteristics. As far as porosity of test specimens is concerned it could be noticed that the total number of pores decreases with gradual aging of powder. However, the size of pores increases. This is visualized in Fig. 4 (b). Powder material which has been used for several production cycles consists of coarser particles. On the one hand powder distribution is more continuous. On the other hand there are probably gaps in the powder bed which cannot be filled by comparatively large particles of recycled powder.

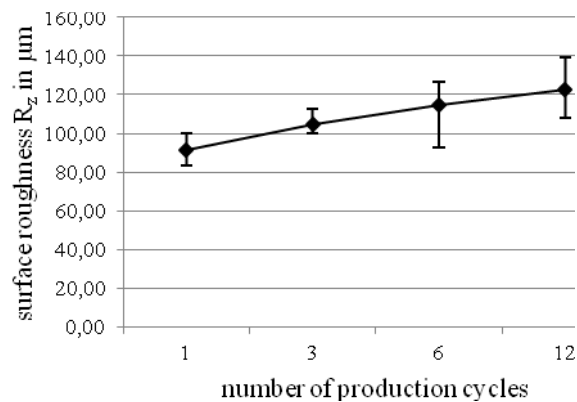


Fig. 5. Average surface roughness of laser melted bulk material

The effect of aging processes of Ti-6Al-4V powder on surface quality of laser melted parts is shown in Fig. 5. Initially, the test cube features a surface roughness of $R_z = 91.58 \mu\text{m}$ in building direction. Roughness of the surface increases the more the material is sieved and reused in the process. After 12 production cycles a surface roughness of $R_z = 122.71 \mu\text{m}$ could be measured. These results correspond to the determined particle size distribution which shows a higher proportion of larger particles of recycled powder material. These large particles melt to the exposed part and attach to its surface which in consequence gets rougher.

Further studies revealed that also hardness and mechanical properties of laser melted bulk material change after powder has been recycled for several times. Hardness of laser melted test cubes amounts to 374 HV1 after 12 cycles. As to the value of 361 HV1 of specimens manufactured using virgin Ti-6Al-4V powder a slight increase in hardness could be ascertained. Powder material is exposed to oxygen of the ambient atmosphere during the removal of parts out of the building chamber as well as during the sieving process. It is assumed that the powder oxidizes and that oxygen atoms are trapped in the melt as the material is liquefied during the process. This would lead to harder laser melted bulk material.

Ultimate tensile strength of test specimens is in the range of 1030 MPa to 1101 MPa. The lowest value of 1030 MPa could be determined for tensile specimens manufactured using virgin Ti-6Al-4V material. Initially, higher values for ultimate tensile strength could be observed with increasing density of parts manufactured using recycled powder material. However, ultimate tensile strength decreases as the size of pores in the microstructure increases with proceeding aging of the material. Large pores which are located close to the surface of the specimen lead to stress concentrations and could cause premature failure. After the decrease to 1072 MPa after six production cycles the values for ultimate tensile strength remain nearly constant.

4. Conclusions

The aim of the investigations discussed in this paper was to gain insight into aging processes of Ti-6Al-4V powder material and to identify possible interdependencies between powder used for laser melting and material properties of manufactured parts. Therefore, results of powder analysis and measured bulk material properties were correlated.

Concluding it can be assessed that properties and condition of Ti-6Al-4V powder material change during use. Due to multiple recycling Ti-6Al-4V powder coarsens and particle size distribution is enlarged. In consequence apparent density increases and flowability is improved.

Relating these findings to material properties of laser melted components it could be noticed that there are non-negligible effects of aged powder on density, porosity, surface roughness, hardness and mechanical strength of bulk material. It could be determined that porosity of laser melted test specimens is reduced by 0.06 percentage points after 12 cycles. This corresponds to a decrease of about 54% in relation to the initially measured porosity value. The reduction in porosity of the part is due to better powder flowability which leads to a more consistent and uniform distribution of powder material on the building platform. However, the wider particle size distribution not only favors flowability but also causes an increase of surface roughness by approximately 33% related to the initially determined value because of existence of comparatively large powder particles.

Further studies on basis material used in laser melting are necessary to completely understand the behavior of Ti-6Al-4V powder material during processing. This understanding is essential to improve and optimize powder transport and powder storage as well as powder handling during the process of laser melting.

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