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Pal, S., Finšgar, M., Gotlih, J., Brajlih, T., Banerjee, P., Yapar, Ö., Lojen, G., Bončina, T., Drstvenšek, I.

ANALYZING PROPERTIES OF SEMI-MOLTEN POWDER GRANULES IN LASER POWDER BED FUSION

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Abstract: *The research focused on the properties of partially fused powder particles and their possible effects on the mechanical and corrosive properties of products produced by Laser Powder Bed Fusion (LPBF). Scanning electron microscopy was used to investigate surfaces and pores, both for adherent particles and for particles completely enclosed in the molten bath. The study focused on Ti-6Al-4V, considering powder particles between 10–45 µm and a layer thickness of 25 µm. Different combinations of laser powers and scanning speeds were chosen to investigate the melting, mixing and solidification behavior. The analysis revealed that certain partially melted powder particles caused microcracks and increased the corrosive surface area, which showed distinct microstructures compared to the core area of the product. The investigation identified fully integrated particles and mostly melted particles by microstructure analysis. The results suggest potential innovative applications in complex product designs where the removal of these particles is a challenge, as well as in bone implantation to improve osseointegration.*

Key words: adhered powder particle, partially melted particle, titanium alloy, porosity, microcrack, microstructure, selective laser melting.

Analiza svojstava polurastavljenih granula praha u fuziji laserskog praha. *Ova studija je istraživala svojstva delimično rastopljenih čestica praha i njihov potencijalni uticaj na mehanička i korozivna svojstva proizvoda proizvedenih postupkom Laser Powder Bed Fusion (LPBF). Koristeći skenirajuće elektronske mikroskopske slike površina i pora, analizirani su procesi kada su se čestice spajale i njihove mikrostrukture, čak i kada je čestica bila potpuno zatvorena u bazenu rastopljene mase. U ovoj studiji ispitani su metalni splav Ti-6Al-4V, pri čemu je opseg veličine čestica praha bio između 10 i 45 µm, a debljina sloja izgrađenog proizvoda iznosila je 25 µm. Istraživane su različite kombinacije laserskih snaga i brzina skeniranja u odnosu na karakteristike topljenja, spajanja i stvrdnjavanja materijala. Nekoliko delimično rastopljenih čestica praha dovelo je do pojave mikropukotina i povećanja korozivne površine. Dodatno, njihove mikrostrukture se razlikovale od onih u jezgru proizvoda. Analizom mikrostrukture pronađene su kako potpuno integrisane, tako i uglavnom rastopljene čestice. Dobijeni rezultati ukazuju na potencijalne inovativne primene za kompleksno dizajnirane proizvode, gde je teško ukloniti ove čestice, kao i za primenu u oblasti implantacije koštanog tkiva sa ciljem poboljšanja osteointegracije.*

Ključne reči: *prilepljena čestica praha, delimično istopljena čestica, legura titanijuma, poroznost, mikropukotina, mikrostruktura, selektivno lasersko topljenje.*

1. INTRODUCTION

Additive Manufacturing (AM) stands out as a premier manufacturing process within the realm of Industry 4.0 [1]. Its hallmark lies in providing unparalleled manufacturing flexibility, particularly for custom production [2]. By employing a layer-by-layer fabrication process, AM has the capability to translate a Computer-Aided Design (CAD) model into a tangible product [3].

The versatility of AM extends across a vast spectrum of materials. It surpasses conventional limitations, enabling the production of refractory and highly flammable metals with remarkable ease [4]. Additionally, AM facilitates in-situ alloying of metals, even those with high melting points and strong chemical affinities [5]. Such capabilities have rendered AM immensely attractive for manufacturers seeking to fabricate components for a myriad of applications spanning aerospace, automotive, mold making, and biomedical fields [6], [7].

While various AM technologies cater to the production of metal parts, Laser Powder Bed Fusion (LPBF), commonly known as selective laser melting, emerges as the preferred choice among manufacturers [8]. This technology's prominence stems from its precision, reliability, and ability to produce intricate and high-quality metal parts by selectively melting and fusing fine layers of powder using a laser.

LPBF's efficacy in manufacturing complex geometries and functional components has positioned it at the forefront of AM methodologies, earning it a pivotal role in numerous industrial sectors seeking advanced and customized production solutions.

In the Laser Powder Bed Fusion (LPBF) process, metallic powder particles pass through a sequence of

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melting, mixing and solidification phases. This happens track by track and layer by layer to form the desired component [9]. During this process, numerous powder particles adhere both to the surface and in the pores of the finished product, which poses a challenge, especially for parts with complicated designs [8].

The adhesion of the powder particles to the part is caused by their partial melting during the manufacturing process, making it difficult to remove them, especially for complex geometries [10]. It is noteworthy that the maintenance of these particles on the implant surface is crucial to improve osseointegration, a critical factor in biomedical applications [11]. However, despite this requirement, these adherent particles can contribute to surface corrosion and the formation of microcracks, which can jeopardize the integrity of the component [9].

One of the main problems associated with these adherent particles is their altered microstructure and bonding to the component. As they are partially melted, their bonding properties and microstructural composition differ from those of the core part [12]. These differences in microstructure and bonding could affect the mechanical properties and integrity of the manufactured part.

Strategies to overcome these challenges include the optimization of process parameters to minimize powder adhesion, the development of post-processing techniques for effective particle removal, and the exploration of surface treatments to mitigate corrosion risks without compromising osseointegration or mechanical integrity.

Titanium alloys, in particular Ti-6Al-4V, are among the most attractive alloy groups as they can be easily machined and processed using Laser Powder Bed Fusion (LPBF) technology. Ti-6Al-4V has an exceptional strength-to-weight ratio and high corrosion resistance [13]. Due to its high biocompatibility, it is widely used for biomedical prostheses, especially for load-bearing and permanent bone implants [14].

LPBF has proven to be the best manufacturing process for parts made of Ti-6Al-4V alloy due to several properties of this alloy. First, Ti-6Al-4V has a high melting temperature, which is well compatible with the high-energy laser melting process of LPBF. In addition, the alloy's high chemical affinity combined with its low thermal conductivity makes it particularly suitable for LPBF [15]. These properties contribute to the fact that the alloy can be successfully produced using the LPBF process. It enables the production of complicated and durable components with high accuracy and precision.

Compared to conventional casting processes, LPBF offers significant advantages and more controllable metallurgical properties in the production of Ti-6Al-4V components [16]. This additive manufacturing method enables better control of microstructural properties, lower residual stresses and the production of parts with superior mechanical properties, including improved fatigue resistance and better control of grain structure.

The LPBF process has changed the production landscape for Ti-6Al-4V components due to its ability to produce highly complex geometries and customise materials for specific applications, offering unparalleled advantages over traditional manufacturing methods. This study fills a significant research gap by investigating the influence of adherent and partially fused powder particles produced during Laser Powder Bed Fusion (LPBF) on mechanical and corrosive properties. While the existing literature comprehensively addresses the mechanical and corrosive properties of LPBF-fabricated materials, the specific effects of these adherent particles have not yet been sufficiently explored. The aim of this research is to characterize the properties of these particles and their effects on mechanical properties, including tensile strength, ductility and fatigue resistance, as well as corrosion behavior. Through detailed analysis and standardized test methods, this study seeks to provide valuable insight into the relationship between adherent powder particles and the overall performance and integrity of components manufactured using LPBF. This has critical implications for optimizing the process and improving material properties in additive manufacturing applications.

2. MATERIALS AND METHODS

The samples used in this study were prepared using fully dense Ti-6Al-4V powder grade 23 obtained from Dentaurum GmbH & Co. KG (Germany), characterized by a particle diameter ranging between 5 and 45 µm. The chemical composition of the provided material comprised 90% titanium, 6% aluminum, 4% vanadium, and collectively less than 1% of nitrogen, carbon, hydrogen, iron, and oxygen. Scanning Electron Microscopy (SEM) images in Figure 1 depict the powder particles, showcasing variations in particle size, illustrating the diverse range of particle dimensions present within the material.

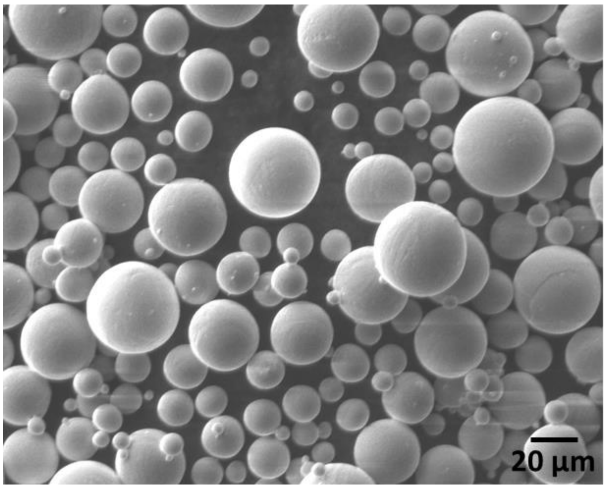

Fig. 1. SEM image of Ti-6Al-4V powder

Numerous cubic samples measuring 8×8×8 mm³ were fabricated by manipulating various parameters such as laser power, scanning speed, hatching distance and volumetric energy density. The laser power ranged from 55 W to 95 W, while the scanning speed varied between 150 mm/s and 1000 mm/s. The hatching distances were adjusted considering the web overlap, increasing the web overlap from 10% to 55% and decreasing the corresponding hatching distance from 0.099 mm to 0.0495 mm. The laser beam with a diameter of 0.110 mm was generated by a Yb:fiber laser from IPG, Germany, and focused with a telecentric F-theta lens perpendicular to the working plane. Despite different parameter configurations, observations indicated partial melting of the powder particles and their adhesion to the vertical surfaces, with a significant reduction in partially melted particles within the pores observed when the laser power was lowered to 65 W and 55 W. The samples were supported by a 2 mm high pedestal with the pedestal plane held horizontally, causing the partially melted particles to accumulate mainly on the four vertical surfaces.

The assessment of sample porosity involved SEM imaging following specific sample preparation procedures. Initially, the samples were ground down by 2 mm and subsequently polished to unveil the surface for porosity analysis. Upon obtaining the porosity images, the polished surfaces underwent an etching process utilizing Kroll's reagent. The preparation of 100 mL of Kroll's reagent involved mixing 2 mL of HF, 6 mL of HNO3, and 92 mL of distilled water, and immersing the exposed surface in this solution for a duration of 30 seconds. Post-etching, the sample's etched surfaces were meticulously examined using field emission scanning electron microscopes (FE-SEM) to observe and analyze the porosity characteristics at a finer scale. This comprehensive process enabled a detailed assessment of porosity levels and distribution within the sample, providing crucial insights into its structural integrity and material composition.

3. RESULTS AND DISCUSSION

The melting characteristics and extent of fusion among powder particles exhibit significant variation. For categorization based on the extent of melting, they've been segmented into three distinct groups: 'less melted,' 'medium melted,' and 'mostly melted' powder particles.

The term 'less melted' denotes powder particles that have undergone partial melting, wherein only a small portion has liquefied. Moderately melted powder particles, on the other hand, encompass specimens where approximately half of the powder composition has undergone melting. Mostly melted powder particles refer to those that have experienced substantial liquefaction, yet they haven't seamlessly integrated into the melt pool. Figure 2 depicts examples showcasing these diverse stages of partially melted powder particles.

All these particles exhibit strong adhesion to the surface or reside within the pores of the final product. In this study, particles displaying minimal melting were disregarded, as they can be effortlessly eliminated from the surface. When such particles persist within the pores, they primarily contribute to the sample's weight without significantly impacting its mechanical or corrosive properties. However, an exception arises if these minimally melted particles come into contact with the corrosive agent subsequent to the corrosion of a larger quantity of the component.

In Figure 2 of the experimental data, two different cases of partially melted particles have been carefully marked and delineated for observation. One of these particles is visibly attached to the periphery of the primary consolidation region, while the other is located in the lowest region of the sample. Although the sample was thoroughly ground and then polished, none of these marked particles showed signs of detachment or removal.

An intriguing observation emerges from this analysis: while the particle adhering to the side exhibits remarkably resilient bonding, the particle located at the bottom appears to be comparatively more susceptible to bond disruption. However, the overarching conclusion from these observations is that both partially melted particles exhibit remarkably robust adhesion to the solidified melt, which is a remarkable result.

This finding emphasizes the considerable interfacial strength that these partially melted particles possessed with the surrounding solidified matrix. Such tenacious bonding could indicate underlying mechanisms controlling the interaction between partially molten particles and the solidifying material, with potential implications for material processing and structural integrity. Further investigation of these binding mechanisms and their effects on overall material properties could provide valuable insights to optimize manufacturing processes and improve material performance in various scientific and industrial fields.

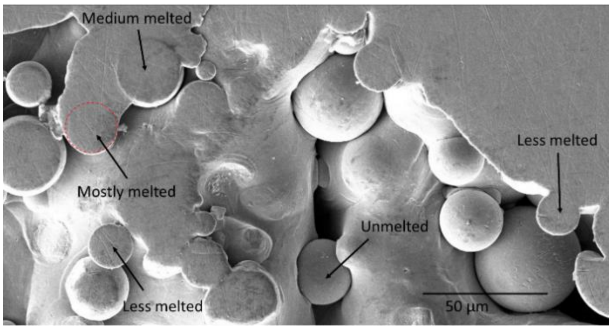

Fig. 2. An ideal example of a pore containing all kinds of partially melted powder particles

A spectrum of partially melted powder particles, each with different characteristics, was observed during the examination of various pores. Among these particles, some have remained benign and have shown no harmful effects, such as the formation of cracks, even under mechanical stress. Others, however, have already triggered microcracks, while a subset have been identified as potential stress concentrators when subjected to mechanical loading.

Figure 3 gives an insight into two categories of mostly fused powder particles. One type has initiated a microcrack, while the other type, despite having no visible cracks, has a high potential for stress accumulation under mechanical loading, as described in reference [15]. In contrast, Figure 2 shows a mostly melted particle that neither induces a crack nor favors the formation of a stress concentration zone.

Figure 4 also shows a less fused particle that does not contribute to the formation of microcracks, nor does it serve as a trigger for crack propagation during product loading. These nuanced observations highlight the different behaviors of partially fused particles and their different effects on the structural integrity and mechanical performance of the final product.

The distinction between these particles, particularly their tendency to either induce cracking or act as stress concentrators under mechanical loading, highlights the complex interplay between the melting of powder particles and its effects on the mechanical behavior of the material. Understanding the role and behavior of these different particle types is of central importance for the elucidation of failure mechanisms, the optimization of manufacturing processes and the strengthening of material designs against structural weaknesses.

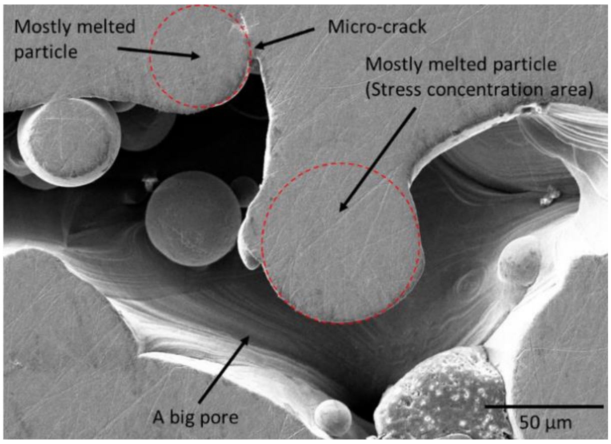

Fig. 3. A pore containing mostly melted powder particles, one of which has produced a microcrack, while the other can accumulate stress under mechanical loading

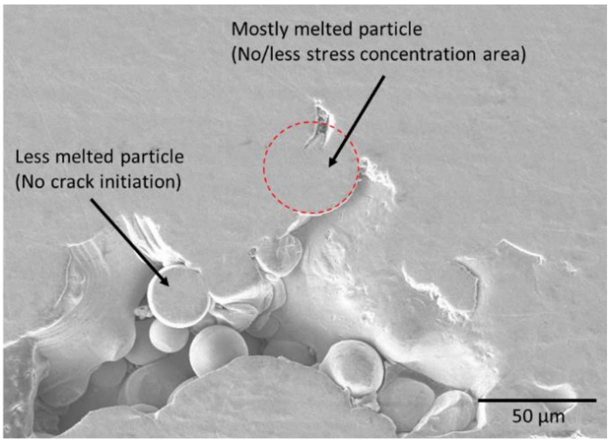

Fig. 4. A pore containing a mostly melted and a less melted powder particle without a crack

The formation of partially melted powder particles is due to various mechanisms. One of these mechanisms is explained in Figure 5. If the melting baths of successive tracks are not sufficient to melt all the powder material, certain particles may partially melt and adhere to one of the melting baths. This insufficiently melted zone results in an irregularly shaped pore containing partially melted particles. If a predominantly or moderately melted particle rests on this pore, there is a considerable chance that microcracks will form.

A moderately melted particle that is molten and has formed a strong bonding with the molten pool will cool rapidly along the molten pool. During this rapid cooling phase, the melt pools contract much faster than the particle itself, creating stresses at the interface between the melt pools and the particle. Although a particle can expand by thermal expansion while most of its volume is melting, a similar situation can occur during the solidification and rapid cooling phase. These circumstances lead to the formation of a corner point in the pore, which serves as a focal point for crack propagation under external mechanical stress.

This intricate process highlights the complex interplay between thermal dynamics, material properties and rapid solidification kinetics that leads to the formation of partially melted particles and their subsequent influence on pore morphology and crack initiation. Understanding these mechanisms is critical to refining manufacturing processes and developing materials that can withstand the vulnerabilities of these partially melted particles.

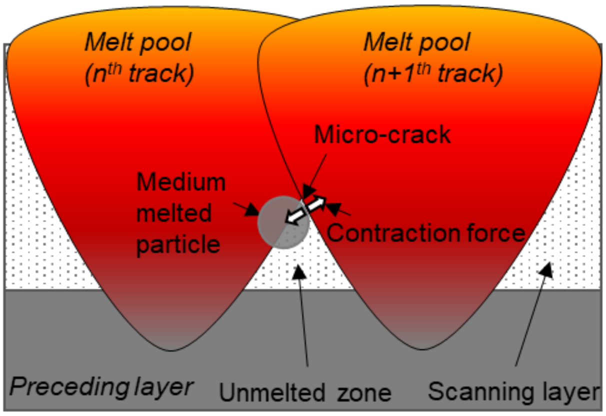

Fig. 5. Formation of microcracks by a medium melted powder particle

Figure 6 shows an alternative mechanism for the formation of mostly melted powder particles. If a powder particle has only recently entered the melt pool, it may not yet be sufficiently molten to fully mix with the molten material. Although fully melted and seemingly integrated into the melt pool, this particle remains distinct within the pool and contributes to the different metallurgical properties. Figure 6(d) shows the microstructure of such a mostly melted particle, which is clearly different from other areas of the component. Consequently, these particles exhibit different mechanical properties and corrosion behavior due to their different metallurgical compositions.

The mechanism underlying the fusion of these types of mostly melted particles with the molten bath is illustrated in Figure 6(a)-(c). Figure 6(a) shows a particle adhering to the surface of the melt and subsequently entering the melt pool. Meanwhile, the vortex in the melt pool tends to flow downward along the vertical surfaces as shown in Figure 6(a) [17]. When the particle enters the pool, it sinks downward with the help of the vortex, as shown in Figure 6(b). However, the rapid solidification of the melt pool occurs from bottom to top [18]. Therefore, the particle may not have enough time to melt thoroughly and bond seamlessly with the melt pool. This results in it remaining as a largely melted particle, as can be seen in Figure 6(c). If it fails to fuse with the molten material in the pool, it leaves behind a distinct microstructure that often has a cellular formation on the surface. Remarkably, this microstructure and grain formation is quite different from the materials that form the melt pool.

This particular phenomenon highlights the profound influence of melt dynamics and rapid solidification kinetics on the formation of mostly melted particles and their impact on microstructural diversity and the resulting material properties. Understanding these mechanisms is essential for predicting and controlling the formation of such particles, which ultimately affect the structural integrity, mechanical properties and corrosion resistance of the material.

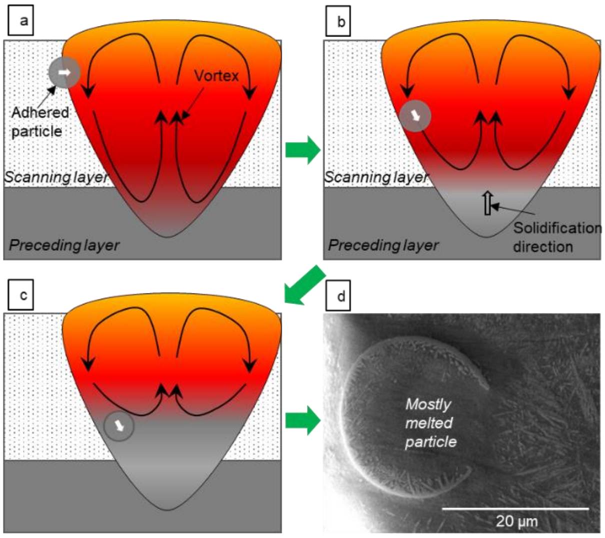

Fig. 6. Mostly melted powder particles entrapment by the melt pool, (a) adhesion of a particle to the melt pool, (b) downward flow of the melt pool with the aid of vortex, (c) cooling due to rapid solidification of the melt pool from the bottom to the top, and (d) microstructure image of a mostly melted powder particle trapped by the melt pool

Numerous partially melted powder particles were observed adhering to the vertical surfaces of the samples. These surfaces are defined as perpendicular to the XY plane, where Z represents the direction of sample buildup. Figure 7 shows the presence of different types of partially fused particles adhering to these vertical surfaces. During the scanning process, the outer surface was covered with powder so that the particles could easily adhere to these surfaces. Once the particles had adhered, they were melted by the heat emitted by the corresponding molten bath.

The degree of melting of the particles depended on the duration between adhesion and the formation of the molten bath and subsequent solidification. A particle that adheres early in the process is more likely to undergo significant melting, possibly taking up most of its volume. Conversely, particles that adhere later could have a moderate or lesser amount of melting. Furthermore, these phenomena could also correlate with the cooling times, which in turn depend strongly on the scanning speed and less on the laser power.

Consequently, a decrease in laser power and scanning speed resulted in a lower porosity of the partially melted powder particles. Although a similar amount of partially fused particles was absorbed from the surface under these adjusted conditions, a higher prevalence of mostly fused particles was observed. This can be attributed to the extended duration that allowed most parts of the particles to melt significantly due to the prolonged exposure to heat.

Understanding the interplay between process parameters such as laser power, scan speed and cooling time and their influence on the degree of particle melting and the resulting particle properties is crucial. These observations highlight the critical role of process control in determining the composition, porosity and distribution of the partially and mostly melted particles, which in turn influences the final material properties and structural integrity.

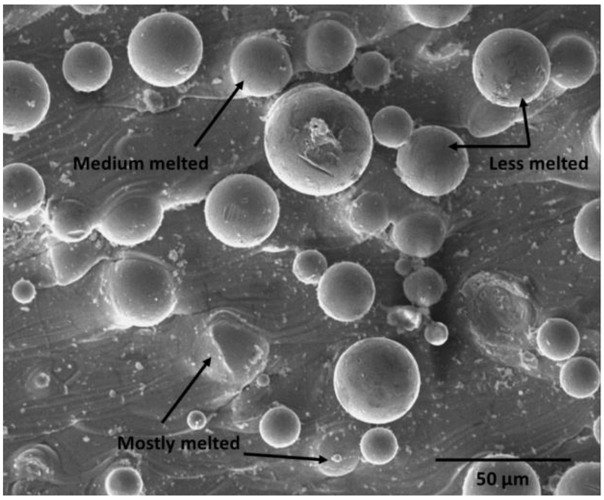

Fig. 7. Several types of partially melted powder particles adhering to the vertical surface of a sample

Figure 8 (d) clearly shows a series of microcracks between the mostly fused particles. In particular, a crack can be seen along the interface between the particles and the molten pool, indicating a clear mechanism for the formation of such cracks, as shown in Figure 8(a)-(c).

When a particle adheres to the melt or enters the melt pool, it is initially affected by the vortex flow, as shown in Figure 8(a). This vortex, which flows downward along the surface of the melt pool, leads to a corresponding downward movement of the particles. At the same time, solidification starts from the bottom and progresses upwards within the melt pool. Consequently, the lower region of the melt pool cools faster compared to the partially melted particles.

This thermal gradient creates a contraction force emanating from the bottom of the melt pool, which causes the particles to maintain a relatively stable position in the middle of the solidifying melt, as shown in the side view of Figure 8(b). In the front view (Figure 8(c)), however, the crack formation is more clearly visible. This contraction force acting between the solidifying material and the surface of the particles leads to a separation or detachment of the molten materials from the particle surface [8]. This detachment mechanism leads to microcracks, as can be seen in the scanning electron micrograph (SEM) in Figure 8(d), which shows a front view of such microcracks.

These intricate observations highlight the crucial role of thermal dynamics and solidification kinetics in controlling the interaction between partially melted particles and the surrounding melt pool. Understanding the mechanisms underlying the formation of microcracks is central to addressing potential structural weaknesses and optimizing manufacturing processes to reduce crack propagation in additively manufactured components.

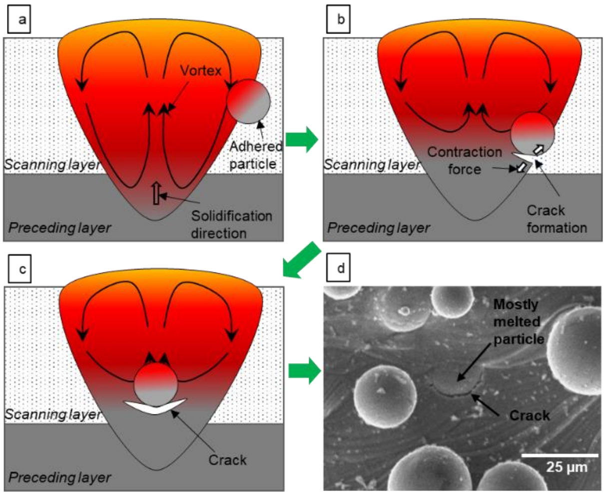

Fig. 8. Mechanism of micro-crack formation by the mostly melted powder particles, (a) adhesion of a powder particle to a melt pool, (b) crack formation – side view, (c) a crack – front view, (d) SEM image of a crack by the mostly melted particles on the vertical surface of a sample

As already shown, the microstructures of the partially melted powder particles differ significantly from those of the main body of the product and thus contribute to different corrosion resistance profiles [12]. This divergence in microstructure results primarily from the significant differences in cooling rates between less melted and mostly melted particles. The extent of melting and the proportion that is integrated into the melt pool significantly influence the microstructural properties.

These nuanced differences in microstructure lead to a wide range of corrosion properties in these particles. Furthermore, the complexity is compounded by the different surfaces as they influence the extent of exposure to the corrosive environment. Consequently, the diversity in microstructural composition and the different surface areas contribute significantly to the different corrosion resistances of these partially fused particles.

The differences in microstructure and surface distribution have a decisive impact on the overall corrosion behavior of the material. Understanding these differences is crucial for predicting and managing the corrosion susceptibility of additively manufactured components and enables tailored approaches to improve their resistance to corrosive environments.

4. CONCLUSIONS

The article provides a comprehensive elucidation of the formation and role of partially fused powder particles in the Laser Powder Bed Fusion (LPBF) process, with particular emphasis on their functions in the production of Ti-6Al-4V alloys. Through a detailed analysis of cross-sectional images, surface images and microstructural investigations, three different types of partially fused powder particles were identified: less fused, medium fused and predominantly fused powder particles. Several important conclusions can be drawn from this study:

i. Partially fused particles predominantly colonize the pores and vertical surfaces of the resulting product, which illustrates their distribution within the manufactured component.

ii. Medium-sized and mostly melted particles, if present, have the potential to form microcracks during the manufacturing process, indicating their role in structural integrity.

iii. These partially fused particles play an important role in the formation of microcracks under mechanical stress, highlighting their importance for structural vulnerabilities.

iv. Partially melted particles can penetrate into the melt pool. However, their incomplete integration into the molten metal leads to significant microstructural differences from the surrounding metal.

v. The different microstructures observed in the different types of partially melted particles directly correlate with their different corrosion resistance profiles, highlighting their influence on the material's susceptibility to corrosion in different environments.

These conclusions shed light on the diverse effects of partially fused particles in LPBF fabricated components by highlighting their influence on mechanical properties, structural integrity, microstructural variations and corrosion resistance. Understanding the role and behavior of these particles is important to drive the optimization and design of additive manufacturing processes to improve material performance and durability in practical applications.

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Authors: **Snehashis Pal[1], Matjaž Finšgar[2], Janez Gotlih[1], Tomaž Brajlih[1], Prabas Banerjee[3], Özkan Yapar[1], Gorazd Lojen[1], Tonica Bončina[1], Igor Drstvenšek[1].** [1]Faculty of Mechanical Engineering, University of Maribor, Slovenia.
[2]Faculty of Chemistry and Chemical Engineering, University of Maribor, Maribor, Slovenia.
[3]Department of Mechanical Engineering, National Institute of Technology Durgapur, Durgapur, India.

E-mail: snehashis.pal@um.si