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Challenges in using powder feedstock for laser powder bed fusion

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Abstract. This paper reviews the main methods for producing and assessing the quality of powder feedstock intended for use in laser powder bed fusion (LPBF). The LPBF process involves the layer-by-layer laser fusion of powder feedstock on the surface of a build plate in accordance with a 3D model. The study examined powder feedstock produced domestically from industrial alloys based on nickel (Inconel 718, EP741NP, AZhK), titanium (VT6, VT6s, VT20), and iron (12Kh18N10T, Fe–Cr–Ni–Co–Mo system). The principal production methods considered are gas atomization, the Plasma Rotating Electrode Process (PREP), and plasma atomization in an inert gas atmosphere, with their respective advantages and limitations described. The most common defects in powder feedstock arising during production and use in LPBF are analyzed, including non-conforming particle size distribution, internal porosity, satellites, changes in bulk density and flowability, fine black particles, increased gaseous impurities, and non-conforming chemical composition. Measures for mitigating these defects and maintaining product quality are proposed. The findings show that achieving stable LPBF results requires regular quality control of powder feedstock to ensure compliance with the requirements specified in applicable standards, including particle size distribution (distribution quantiles d_{10}, d_{50}, and d_{90}), processing characteristics, particle morphology, chemical composition, and moisture content. For certain alloys, when defects occur systematically and cannot be effectively eliminated through process adjustments or post-processing, the most appropriate solution is to change the powder production method.

Keywords: additive manufacturing, powder feedstock, laser powder bed fusion (LPBF), heat-resistant alloys, powder defects, nickel-based alloys, titanium-based alloys, iron-based alloys

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О проблемах применения металлопорошковых композиций в технологии селективного лазерного сплавления

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Аннотация. В работе рассмотрены основные методы производства и контроля качества металлопорошковых композиций (МПК), предназначенных для применения в технологии селективного лазерного сплавления (СЛС). Метод СЛС представляет собой послойное лазерное сплавление МПК на поверхности металлической подложки в соответствии с 3D-моделью. В качестве объектов исследования использованы МПК из промышленных сплавов на основе никеля (Inconel 718, ЭП741НП, АЖК), титана (ВТ6, ВТ6с, ВТ20), железа (12Х18Н10Т, система Fe–Cr–Ni–Co–Mo) отечественного производства. Основными методами их изготовления являются газовая атомизация, плазменное центробежное распыление, плазменная атомизация в среде инертных газов. Приведены основные преимущества и недостатки каждого из представленных способов производства МПК. Рассмотрены наиболее распространенные дефекты МПК, возникающие на этапе их получения и применения в процессе СЛС, такие как несоответствие гранулометрического состава, внутренняя пористость, сателлиты, изменение насыпной плотности и текучести, нагар, увеличение содержания газовых примесей и несоответствие химического состава. Предложены основные способы их устранения для сохранения качества выпускаемой продукции. Установлено, что для получения стабильных результатов в процессе послойного синтеза методом СЛС необходимо проводить регулярный контроль качества МПК на соответствие установленным в нормативной документации требованиям (гранулометрический состав – квантили распределения d_{10}, d_{50} и d_{90}, технологические свойства, форма частиц, химический состав, влажность). В случае возникновения на регулярной основе дефектов, которые затруднительно и/или невозможно устранить, наилучшим решением для некоторых сплавов является смена метода производства МПК.

Ключевые слова: аддитивные технологии, металлопорошковые композиции (МПК), селективное лазерное сплавление, жаропрочные сплавы, дефекты порошков, сплавы на основе никеля, сплавы на основе титана, сплавы на основе железа

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Introduction

The continuous development of powder metallurgy has led to the emergence of a modern method for producing components – additive manufacturing (AM). This technology encompasses techniques for the layer-by-layer fusion or deposition of powder or wire feedstock using high-energy sources such as lasers, electron beams, or electric arcs. The material is fused layer by layer in accordance with a 3D model [1].

The most widely employed AM process is laser powder bed fusion (LPBF), or selective laser melting (SLM), in which layers of powder are selectively fused by a laser on the surface of a build plate (also referred to as the substrate). The production of a component by LPBF typically involves the following stages:

– preparation of the 3D model (slicing, generation of support structures, positioning of the part on the build plate, and setting the LPBF process parameters);

– preparation of the equipment (cleaning, installation of the build plate, and uploading the prepared model file to the control unit);

– execution of the LPBF process;

– removal of the build plate with the fabricated part;

– post-processing of the part (mechanical processing, thermal treatment, and hot isostatic pressing).

Each stage of the process requires close control, as any deviation from the prescribed parameters may adversely affect the quality of the final component [2].

In addition to the stability of the equipment and software, the key factors influencing the quality of the end product include the properties of the powders, the LPBF process parameters, and the precision of the part geometry.

Powder feedstock – referred to in Russian standards as a metal powder composition (MPC) – is defined in GOST R 59035-2020 as a metallic powder formulated as a single composition for use in AM. At both the production stage of the MPC and during the use of powder feedstock in layer-by-layer synthesis, it is essential to control parameters such as particle morphology, particle size distribution, chemical composition, processing characteristics (apparent density and flowability), defects (internal – gas porosity; external – satellites), and moisture content. Any non-conformity of the powder feedstock with the specified requirements can result in diminished quality of the finished product [3–6].

A correctly selected LPBF mode – representing the combination of process parameters – is one of the key factors determining the final result, including the mechanical properties of the part and its compliance with the dimensions specified in the design documentation. For this reason, LPBF parameters are developed for each alloy, and dimensional optimization is carried out for each part. Both of these stages are labor-intensive and are implemented iteratively, with intermediate results followed by adjustments to the process parameters.

The final stage in producing the target component is comprehensive post-processing, involving the selection of the necessary mechanical and thermal operations. At this stage, the part is separated from the build plate, support structures and any residual powder feedstock are removed from its surface and internal channels, and machining is performed to achieve the dimensions specified in the design documentation. Thermal treatment and hot isostatic pressing are used to relieve residual thermal stresses, reduce and/or eliminate characteristic structural defects (such as pores and cracks), and form a microstructure that ensures optimal properties, for example through the precipitation of strengthening phases. The sequence of technological operations is determined individually for each alloy and part type [7–10].

According to the Order of the State Corporation Roscosmos dated 12 November 2021, No. 332, JSC "Kompozit" has been designated as the competence center for additive technologies in the rocket and space industry. In this capacity, it undertakes activities aimed at the development, design, analysis of new materials, as well as the refinement of technological approaches and the provision of services in the field of AM. To date, more than ten grades of industrial alloys have been adapted for LPBF, including EP741NP, AZhK, VZhL12U, VT6, VT6s, VT20, VNL-3, EI712, EP810, EI835, and others [11–13]. The results of this adaptation have shown that the nature of the alloy (chemical composition and processing characteristics) and the quality of the powder feedstock have a decisive influence on the final outcome. It should also be noted that alloys adapted for LPBF cannot always be used in other AM methods due to the specific nature of layer-by-layer synthesis in each process.

The aims of the present study were to:

– review the principal methods for producing and controlling the quality of powder feedstock used in LPBF;

– analyze the requirements for such feedstock;

– examine the characteristic defects in powder feedstock and the measures used to eliminate them.

Experimental procedure

The study examined powder feedstock produced by domestic manufacturers from industrial alloys based on nickel (Inconel 718, EP741NP, AZhK), titanium (VT6, VT6s, VT20), and iron (12Kh18N10T and alloys from the Fe–Cr–Ni–Co–Mo system).

The characteristics of the powder feedstock – both in the as-supplied state and after LPBF – were assessed using standardized methods:

– bulk density in accordance with GOST 19440-94 and GOST R 70907-2023;

– flowability, in accordance with GOST 20899-98 and GOST R 70910-2023;

– particle size distribution (granulometric composition), in accordance with GOST 18318-94 and GOST R 70909-2023;

– particle morphology, in accordance with GOST 25849-83 and GOST R 70908-2023;

– chemical composition, depending on the alloy grade;

– gaseous impurities, depending on the alloy grade.

The microstructure of the powder feedstock and of samples fabricated by LPBF was examined using optical microscopy (OM) with an AxioVert A1 microscope (Carl Zeiss, Germany) equipped with an E3IS PM digital camera (Touptek Photonics, China) for image capture, and by scanning electron microscopy (SEM) with an S-3400N microscope (Hitachi High-Technologies Corporation, Japan).

Results and discussion

Characteristics of powder feedstock: Structure and properties

In most cases, the powder feedstock used in LPBF must meet a standard set of requirements:

– particle size distribution in the range of 10–63 μm (the specification may indicate distribution quantiles d_{10}, d_{50}, and d_{90}, as well as the percentage of particles outside the target fraction);

– flowability of no more than 50 s;

– bulk density of at least 0.5 of the material's density;

– spherical or near-spherical particle morphology;

– moisture content not exceeding 0.01 %;

– chemical composition in accordance with the alloy grade.

If no specific requirement for oxygen content is given, its allowable limit for the particular alloy should be established by collecting statistical data. This is important because, during repeated use (when powder not exposed to the laser is recycled), the oxygen content in the feedstock may increase, leading to degradation of the material's mechanical properties.

Methods of powder feedstock production

The main methods for producing powder feedstock are gas atomization, plasma rotating electrode process (PREP), and plasma atomization in an inert gas atmosphere. The key advantages and disadvantages of each method for producing powder feedstock from nickel-, iron-, and titanium-based alloys are outlined below.

Gas atomization is one of the most widely used and high-throughput techniques for producing powder materials. In this process, the metal is melted in an induction furnace and then atomized with an inert gas such as argon. The method is most often applied to the production of nickel- and iron-based powders.

Its advantages include low production cost, owing to the high process productivity and the straightforward melt preparation stage (heating system and charge material selection); capability to produce a broad range of materials; and narrow particle size distribution.

The disadvantages are: the presence of satellites on particle surfaces; internal gas (argon) porosity; oxidation of particles (in the case of open melting); and presence of a fine particle fraction, which impairs the processing characteristics of the feedstock and requires an additional separation step. Internal porosity forms during atomization when molten droplets entrap argon gas. Satellites occur because finer particles solidify more rapidly, are more easily entrained, and collide with larger particles under the influence of turbulent flows [14].

Plasma rotating electrode process (PREP) uses a cylindrical billet as the feedstock. While the billet rotates at high speed (up to 35,000 rpm), its end face is melted by a high-power direct-current plasma arc. This produces a thin molten film on the billet end, from which droplets detach and spheroidize under surface tension forces. Atomization takes place in an inert gas atmosphere (argon–helium mixture) under overpressure.

This technique is most frequently used to produce nickel- and iron-based powders. Its key advantages are: excellent processing characteristics of the resulting powder; uniform chemical composition; low levels of gaseous impurities; high productivity; and minimal internal and external defects. The latter results from the specifics of the centrifugal atomization process: the relatively low cooling rate allows each droplet to fully spheroidize before solidification, while the absence of turbulent flows reduces the formation of satellites [14].

The main drawbacks of PREP are: higher cost of powder feedstock, since the starting cylindrical billet requires several production stages (melting, machining) and must meet strict quality requirements (minimal run-out, uniform chemical composition, absence of defects such as cracks and cavities); low yield of the target particle size fraction; and a limited range of industrial alloys suitable for the method [15].

Plasma atomization in an inert gas atmosphere employs wire feedstock with a diameter of up to 5 mm. This technology is used primarily for titanium-based powders and selected nickel-based alloys. Its advantages include homogeneous chemical composition; narrow particle size distribution; low levels of internal and external defects; and high processing performance.

Its disadvantages are: a restricted range of alloys (limited to those available in wire form); lower productivity compared with the methods described above; and relatively high production cost [16].

Plasma spheroidization also merits mention. In this process, powders are treated in a thermal plasma jet. While not an independent production method, it is used to spheroidize powders with angular or irregular particle shapes, rendering them suitable for LPBF. Plasma treatment also reduces the occurrence of satellites, thereby improving the processing properties of the feedstock. However, it may alter the particle size distribution and increase the proportion of fine particles, which adversely affects flowability. To remove the fine fraction, additional separation steps are performed, such as ultrasonic washing.

Defects in powder feedstock: Causes, formation mechanisms, and prevention measures

Non-conforming particle size distribution. Deviation in the particle size distribution of the powder feedstock from that originally used to establish LPBF process parameters changes the overall energy input and disrupts heat transfer. This can promote the formation of cracks and warping in the material (Fig. 1, *a*). Such deviations are cumulative, developing through repeated reuse of the same batch of powder [17]. A reduction in the proportion of fine particles impairs the deposition of powder feedstock onto the build plate, leading to a decrease in the density of the deposited layer. Laser exposure on a less densely packed layer leads to local overheating, increased thermal stresses, and the formation of defects such as cracks (Fig. 1, *b*). This effect is most pronounced when building parts of complex geometry and/or large size.

The issue can be addressed by adjusting LPBF parameters and/or blending in powder of a specific particle size to achieve the required particle size distribution with the target quantiles d_{10}, d_{50}, and d_{90}.

Internal porosity. This defect originates during powder production through entrapment of process gas, most commonly in gas atomization. Using feedstock with internal gas porosity in LPBF results in its transfer to the consolidated material and in uneven fusion, producing so-called fine black particles (Fig. 2). Porosity can be reduced by optimizing fusion parameters and/or

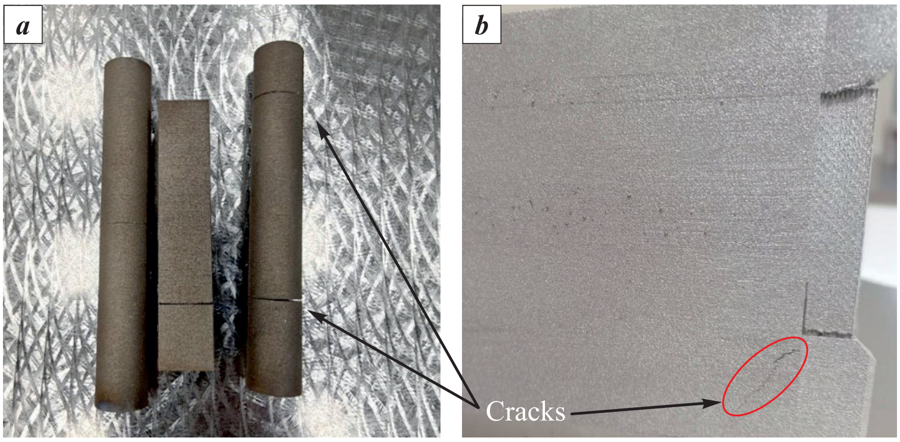

Fig. 1. Appearance of surface defects in a VT6 witness samples (***a***) and in a VT20 workpiece (***b***)

Рис. 1. Внешний вид поверхностных дефектов в образцах-свидетелях из сплава ВТ6 (***a***) и заготовке из сплава ВТ20 (***b***)

using powder with minimal gas porosity, for example by selecting an alternative production method.

Satellites. As noted earlier, satellites are characteristic of gas atomization. A high proportion of satellite-bearing particles reduces the processing performance of the powder and increases the formation of fine black particles during layer-by-layer fusion. The formation of these particles can be mitigated to some extent by optimizing fusion parameters. The satellite content can be lowered through additional sieving and thermal plasma treatment. However, these steps increase powder preparation complexity and the cost of the final product.

Bulk density. Bulk density should be carefully considered when selecting powder feedstock. It should be as close as possible to the theoretical maximum and comparable to that of powders from leading suppliers. Lower bulk density values are generally caused by higher internal porosity and/or changes in particle size distribution. Using powder with reduced bulk density in LPBF can lead to additional defects in the consolidated material and contribute to the formation of fine black particles.

This issue can be resolved by additional classification and/or blending powder of a specific particle size to restore the required particle size distribution with the target d_{10}, d_{50}, and d_{90} values. It is also advisable to verify the stability of the powder production process.

Flowability. Reduced powder flowability can result from changes in particle size distribution, an increased proportion of defective particles, or – in rare cases – elevated moisture content due to improper storage. Poor flowability increases equipment preparation time and can clog the powder recycling system, if present. Studies have also shown that powders with poor flowability spread less effectively across the build plate, affecting both the recoating process and layer uniformity [17–19].

Flowability can be improved by additional classification and/or adjusting the particle size distribution.

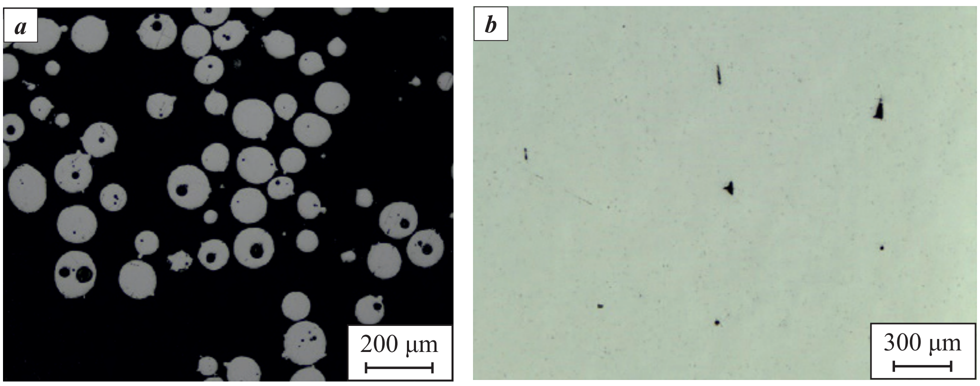

Fig. 2. Microstructure of powder (***a***) and LPBF-fabricated Inconel 718 material (***b***) with internal porosity

Рис. 2. Микроструктура МПК (***a***) и СЛС-материала из сплава Inconel 718 (***b***) с внутренней пористостью

If moisture is the cause, vacuum drying should be carried out.

Fine black particles. The term refers to fine, dark-colored particles generated during layer-by-layer fusion. Their accumulation clogs the filtration system and contaminates optical components, and when deposited on previously fused layers, they can cause defects or even halt the build (Fig. 3). Increasing the shielding gas flow can remove fine black particles from the melt pool area but also displaces fine powder particles into the filtration system. Reducing the fine fraction mitigates their formation but alters the feedstock particle size distribution. Observations indicate that powders with high internal porosity and a high proportion of satellites tend to generate more fine black particles. Adjusting LPBF parameters has not produced significant improvements. Further research is planned to investigate their formation mechanisms in powders from different alloys.

Gaseous impurities. The results of this study, together with literature data, indicate that under proper storage conditions, oxygen uptake by powder feedstock is negligible. In titanium-alloy powders, a slight increase in oxygen content during storage has been observed, but it remains within the permissible limits for the given alloy grade. Oxygen concentration increases when environmental conditions are not adequately controlled, as well as when operating equipment with a build chamber temperature above 500 °C. At present, most industrial LPBF systems do not have this capability. Notably, adding fresh powder feedstock to recycled powder slows the rate of oxygen uptake – a feature typical of titanium-based alloys [18; 19].

The use of powder feedstock containing pre-oxidized particles results in the transfer of oxide phases into the LPBF-fabricated material, reducing its mechanical properties, particularly ductility [19]. The oxygen content does not decrease during the LPBF process. Fig. 4 shows a typical microstructure of powder feedstock particles from an Fe–Cr–Ni–Co–Mo alloy system containing oxide inclusions. Powders with such defects are not suitable for LPBF.

Scale factor. When optimizing LPBF parameters using small test samples – most often cubes measuring 10×10×10 mm – the geometry of larger workpieces must be considered. Due to the inherent characteristics of the LPBF process, thermal stresses accumulate during layer-by-layer fusion, which can lead to warping and cracking (Fig. 5). As a result, manufacturing a workpiece with complex geometry may require several iterations, each followed by adjustments to the 3D model and LPBF parameters. Currently, there are no software tools capable of fully minimizing this opti-

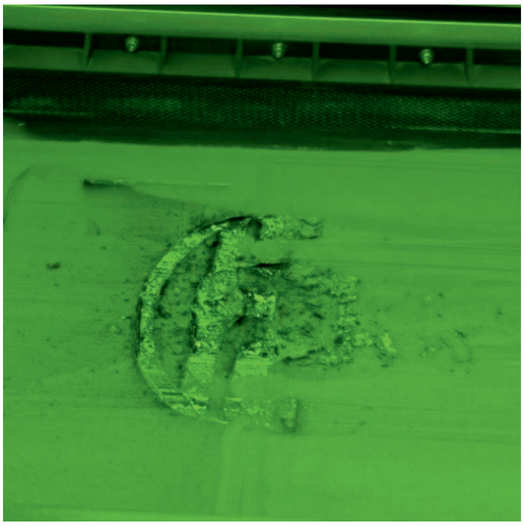

Fig. 3. Appearance of an Inconel 718 workpiece with accumulated fine black particles, which led to interruption of the LPBF process

Рис. 3. Внешний вид заготовки из сплава Inconel 718 с образовавшимся нагаром, что привело к остановке СЛС-процесса

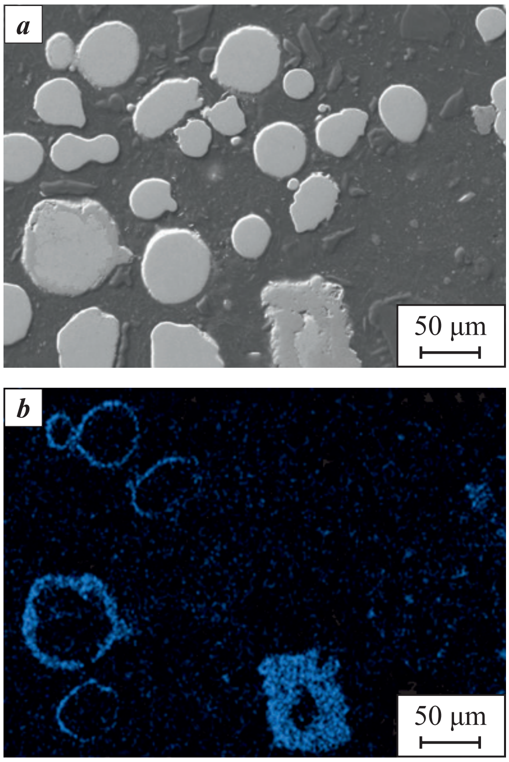

Fig. 4. Microstructure of oxidized powder feedstock particles from an Fe–Cr–Ni–Co–Mo alloy system (***a***) and oxygen distribution map (***b***)

Рис. 4. Микроструктура окисленных частиц МПК сплава системы Fe–Cr–Ni–Co–Mo (***a***) и карта распределения кислорода (***b***)

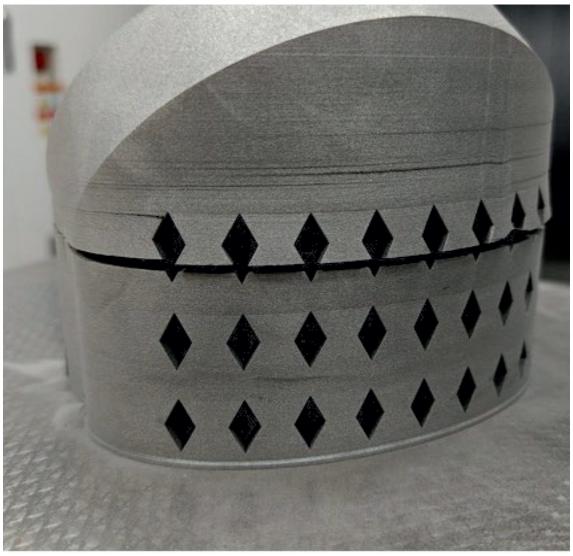

Fig. 5. Appearance of a VT6 workpiece showing delamination defects caused by high thermal stresses

Рис. 5. Внешний вид заготовки из сплава ВТ6 с дефектами в виде расслоения вследствие высоких термических напряжений

mization process, making operator expertise a critical factor.

Chemical composition. Analysis of the initial nickel-, titanium-, and iron-based powder feedstock and of the materials produced from them revealed no changes in the content of the principal alloying elements. Literature sources report that evaporation can occur when the process is carried out with excessive overall energy input. In this study, it was found that the elements most prone to evaporation are the low-melting constituents Mg, Zn, and Al [20]. For this reason, the overall chemical composition is monitored less frequently than gaseous impurities.

Conclusion

The present analysis has demonstrated that detecting all of the defect types considered – porosity, cracks, oxide inclusions, and others – requires regular quality control of powder feedstock to monitor for trends indicating degradation of its properties. This involves assessing particle size distribution, processing characteristics (bulk density and flowability), particle morphology, and chemical composition, both in the as-supplied state and after layer-by-layer fusion.

Consistently using high-quality powder feedstock ensures stable process outcomes. When defects such as porosity or fine black particles occur systematically, or when the feedstock exhibits poor processing performance that cannot be effectively corrected through LPBF parameter adjustment or post-processing, the most appropriate solution for certain alloys is to change the powder production method.

There is also a growing trend towards adapting industrial alloys for additive manufacturing. However, not all materials are suitable for layer-by-layer fusion due to the specific features of their chemical composition. Since additive manufacturing is closely related to welding, alloys with limited weldability are generally unsuitable for layer-by-layer fusion.

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