

Technical Article**Additive Manufacturing in the Development of Ni-Base Superalloys****Kh. Momen, Z. S. Seyedraoufi*, M. Abbasi***Advanced Materials Engineering Research Center, Karaj Branch, Islamic Azad University, Karaj, Iran.**Received: 02 June 2024 - Accepted: 21 November 2024***Abstract**

In this research, laser-based additive manufacturing (AM) of Ni-based superalloys has been evaluated. By reviewing past research and recognizing the challenges and benefits of additive manufacturing, the future of this path can be well described. For this purpose, it is very important and useful to know the target industries, the resulting microstructure, the relationship between the microstructure and the mechanical properties of the final part. In addition, the ways to prevent possible defects of this process are summarized in the report. This report tries to introduce this process in the direction of less damage to the environment and available resources. To be introduced as an environmentally friendly method for manufacturing parts needed for energy supply and transmission. In the future, more reports of this process will be provided by the authors of this review article.

Keywords: Additive Manufacturing, Ni-base, Superalloy, Microstructure.

1. Introduction

Ni-based superalloys are high-performance materials developed for extreme conditions, particularly where high temperature, oxidation resistance, and mechanical strength are crucial [1-3]. These materials have found extensive use in industries such as aerospace, power generation, and marine applications, especially in gas turbine engines, where components are subject to high temperatures and stresses [4,5]. Ni-based superalloys often include alloying elements like Cr, Co, Mo, Ti, Al, and others in smaller amounts to enhance specific properties. They are unique due to their excellent stability at high temperatures (up to about 1,100°C) and exceptional resistance to oxidation, hot corrosion and high temperature corrosion [6-8]. This stability is primarily due to their microstructure, which typically consists of a FCC matrix phase, known as the γ phase, strengthened by a secondary precipitate phase known as γ' , which is an intermetallic compound of Ni and Al (Ni₃Al). γ' phase provides the alloy with high-temperature strength and stability, as it resists deformation and dislocation movement [1,9]. Another phase γ'' , is common in Ni-Fe-based superalloys, offering extra hardening in some specialized applications [10]. Ni-base superalloys have special advantages compared to other high temperature alloys. These special advantages depend on their microstructure and physical metallurgy. Among these advantages and their reasons, the following can be mentioned:

- **High-Temperature Strength:** The γ and γ' phases provide superalloys with exceptional strength at elevated temperatures, allowing them to perform under high stress [11].

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- **Oxidation and Corrosion Resistance:** Elements like Cr and Al form a protective oxide layer that prevents further oxidation and corrosion, especially at high temperatures [12].

- **Fatigue and Creep Resistance:** Ni-base superalloys maintain mechanical strength over prolonged periods of high stress and temperature, which is essential for applications like turbine blades, which experience constant cyclic loading [13].

- **Thermal Stability:** Their microstructure remains stable at high temperatures, unlike many other materials that might undergo phase transformations or lose integrity [14].

- **Tailored Properties:** Through the addition of elements like W, Mo, and Co, superalloys can be fine-tuned for specific applications, achieving the desired balance between strength, ductility, and resistance to thermal deformation [15-17].

Having all the above advantages is not so easy for artisans and researchers. The users of Ni-base superalloys to have the mentioned advantages are faced with several problems such as the following:

- **Cost of Raw Materials:** Ni and the alloying elements used in superalloys, such as Re, Hf, Ta, Nb and Pt are expensive, increasing production costs.

- **Manufacturing Intricacy:** Producing superalloys requires advanced processes like vacuum induction melting (VIM) and powder metallurgy to avoid contamination and ensure homogeneity.

- **Machining and Shaping:** Ni-base superalloys are difficult to machine due to their strength and hardness, especially at high temperatures. Conventional machining can lead to excessive tool wear, necessitating the use of specialized cutting tools or even laser machining [18, 19].

- **Thermal Processing Challenges:** To achieve the necessary microstructure, superalloys require

complex heat treatments, such as solution annealing and aging. Precise control of temperature and cooling rates is essential to obtain the desired properties, increasing manufacturing complexity [20].

- Sensitivity to Defects: Superalloys are sensitive to microstructural defects, such as cracks, voids, or inclusions, which can compromise their high-temperature performance. Stringent quality control measures, such as X-ray inspection, are necessary, adding to production costs [21].

- Environmental Impact: The extraction and processing of alloying elements, as well as the energy-intensive production methods, have an environmental footprint, which is a concern for sustainable manufacturing.

Ni-based superalloys continue to be invaluable in high-performance applications despite these challenges, thanks to ongoing advancements in production methods and alloy design that aim to improve their efficiency and reduce their environmental impact.

2. New Processing Methods of Ni-base Superalloys

Recent advancements in the production of Ni-based superalloys have focused on improving their mechanical properties, reducing production costs, and addressing environmental concerns. Some of the newer methods include additive manufacturing (AM) / 3D printing, powder metallurgy (PM), spark plasma sintering (SPS), and laser cladding.

2.1. Additive Manufacturing (AM) / 3D Printing

Additive manufacturing, particularly laser powder bed fusion (LPBF) and electron beam melting (EBM), has emerged as a promising method for producing Ni-based superalloy components [22-25]. AM allows for complex geometries that are challenging or impossible with traditional casting and forging methods. It also reduces material waste, as it builds components layer by layer, rather than removing material from a block. AM allows for local microstructural control, which can optimize properties in critical areas of a component. The process can introduce defects like porosity and residual stress, which affect mechanical properties. Post-processing techniques, such as hot isostatic pressing (HIP) and heat treatment, are often required to improve structural integrity [23, 26-28].

In general, the HIP process leads to improving the performance of SLM parts. Through diffusion control, the size of γ' precipitates will be reduced because hot pressure will lead to the reduction of interatomic distances and hinder growth. In Fig. 1., the SEM images show how the γ' precipitates did not grow much after HIP and subsequent aging. Bassini et al. [29], showed in a research how HIP

leads to the elimination of SLM process defects. In the SEM image of Fig. 2., the results of their work are shown, how the cracks are fixed.

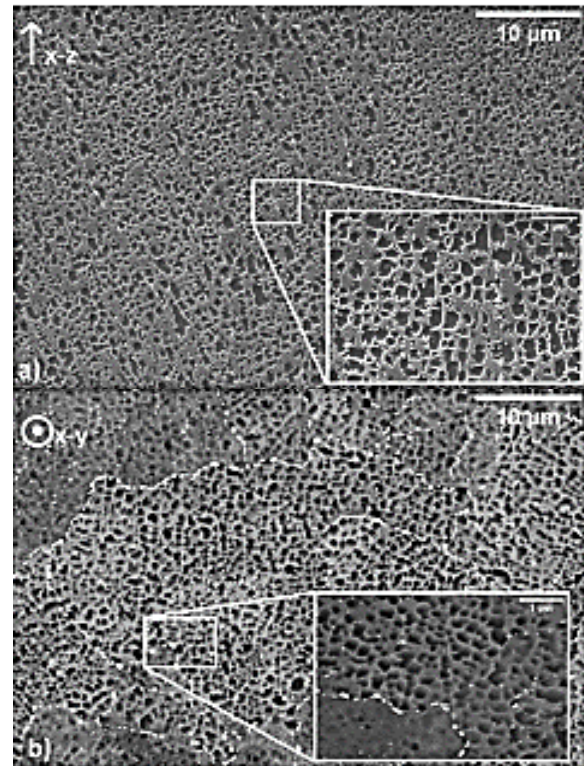


Fig. 1. Microstructures of the SLM CM247lc observed in the x-z plane (a) and in the x-y plane (b) after being HIPped [29].

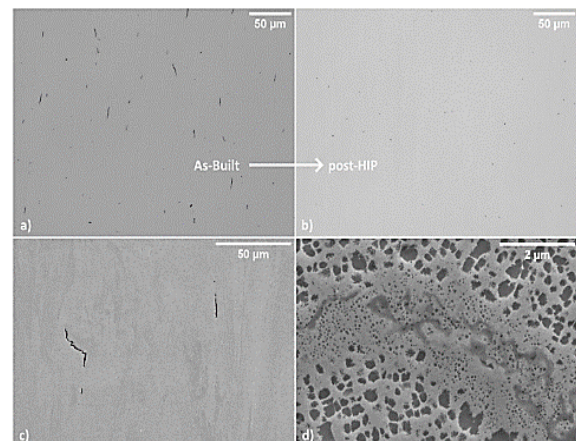


Fig. 2. The application of the HIP treatment closes the majority of the defects. Pictures a) and b) were taken with an optical microscope and clearly demonstrate the elimination of the cracks. Only small pores are retained. Picture c) shows cracks using the SEM, while d) shows how a crack looks like after its closure due to HIP application [29].

2.2. Powder Metallurgy (PM)

Powder metallurgy (PM) has been refined to create superalloy parts with high purity and precision. It is

often used for turbine discs and other critical parts that need excellent fatigue resistance. Metal powders are produced via gas or plasma atomization, compacted, and then sintered to form solid parts. Techniques such as HIP help eliminate porosity, improve density, and enhance material properties. PM allows for uniform distribution of alloying elements, which helps achieve consistent mechanical properties. PM also reduces material waste. Producing high-quality powders can be costly, and controlling powder purity is critical to prevent impurities that may lead to defects [30, 31].

2.3. Spark Plasma Sintering (SPS)

Spark plasma sintering is a relatively new technique used in the powder metallurgy process to densify metal powders. In SPS, an electric current is applied directly through the powder, generating heat and causing particles to fuse. This method reduces the processing time and temperature compared to conventional sintering. SPS can produce dense materials with a fine-grained microstructure, which enhances strength and fatigue resistance. It also reduces production time. The scalability of SPS for larger or more complex parts is still being explored, and controlling uniform heating throughout the part can be challenging [34].

In rapid heating, the diffusion paths change and the diffusion rate increased for this reason, the type of precipitation also changes and improves [35]. SPS is a rapid heating method. Fig. 3. illustrates the formation process of carbides during sintering as prior particle boundary. The sintering process facilitates the segregation of carbide composition elements such as C, Ti, and Nb due to their high diffusion coefficients. These elements tend to segregate at the sintered neck. Owing to the lower nucleation energy of carbides compared to oxides at the interface, MC type carbides precipitate in the sintered neck [36].

Despite the less apparent presence of prior particle boundary, a certain amount of oxy carbide was observed within the matrix. This can be attributed to the sintering mechanism, where the pre-alloyed powder experienced plastic deformation prior to solid-phase sintering.

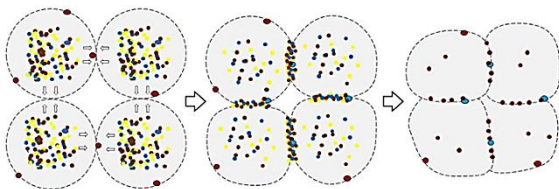


Fig. 3. The evolution of carbides as prior particle boundary [36].

As a result, the particle boundaries of the powder were partially disrupted. Since the sintering temperature was relatively low, the formation of the

sintering neck occurred gradually. Consequently, the diffusion of carbide-forming elements towards the powder boundaries was incomplete, leading to nucleation within the matrix [36]. In fact, the change in the rate and type of heating caused the inside of the grain to be a suitable place for the formation of primary carbides (MC), just like the grain boundaries in FGH96 Ni-base superalloy.

2.4. Laser Cladding

Laser cladding is used to apply a layer of Ni-based superalloy onto substrates, which can extend component life and allow for complex repairs. A laser beam is used to melt metal powder or wire as it is deposited on a substrate, creating a protective layer with enhanced resistance to wear, corrosion, or heat. Laser cladding is precise and minimizes the heat-affected zone, preserving the properties of the underlying material. It is particularly useful for extending the life of expensive components, like turbine blades. Laser cladding can introduce residual stresses, and the bonding between the cladded layer and substrate must be carefully controlled [37].

These new production methods enhance the capabilities of Ni-based superalloys, but they also come with unique challenges and costs. By continuing to develop and refine these methods, manufacturers can produce Ni-based superalloys that are more efficient, reliable, and suited to the demanding conditions of modern applications, from aerospace to power generation. Meanwhile, AM has been a revolution in the field of manufacturing gas turbine parts made of Ni-based superalloy. That's why it is necessary to know inside it

3. AM of Ni-Base Superalloys

AM also known as 3D printing has revolutionized the production of Ni-based superalloys by enabling complex geometries, reducing material waste, and offering new ways to tailor material properties. It is particularly transformative for industries like aerospace, energy, and automotive, where high-performance Ni-based superalloy components are essential. Several AM methods are suitable for Ni-based superalloys. The most widely used techniques include:

- Laser Powder Bed Fusion (LPBF) that also known as selective laser melting (SLM), LPBF uses a high-powered laser to selectively melt fine alloy powder in a bed layer-by-layer. This method is popular for producing small to medium-sized components with intricate designs, such as turbine blades [24,26].
- Electron Beam Melting (EBM) similar to LPBF, EBM uses an electron beam rather than a laser to melt metal powder. This method occurs in a vacuum, which reduces oxidation and allows for a faster build rate. EBM is commonly used for applications requiring high density and strength [25].

- **Directed Energy Deposition (DED, in DED,** powder or wire feedstock is melted by a laser, electron beam, or plasma arc as it is deposited onto the surface, building the structure layer by layer. DED is often used for larger components, repair applications, and cladding [38].

- **Binder Jetting,** in this process, a liquid binder is selectively deposited onto a powder bed to create a green part, which is later sintered. Although less common for Ni-based superalloys, binder jetting can be cost-effective for producing parts with intricate geometries that do not require extreme high-temperature performance [39].

AM offers several unique advantages for producing Ni-based superalloy components:

- **Complex Geometries:** AM allows for the creation of complex shapes, lattice structures, and internal channels as cooling holes, which are difficult or impossible to achieve with conventional methods for gas turbine fabrication. This capability is particularly valuable for parts like turbine blades, where aerodynamic optimization is critical.

- **Material Efficiency:** Traditional manufacturing methods, like forging or casting, often involve significant material waste. In contrast, AM builds parts layer by layer, reducing material waste and lowering the cost of expensive Ni-based superalloys.

- **Localized Microstructure Control:** AM can be used to control microstructure locally within a part, optimizing properties in specific regions that undergo higher stress or temperature exposure.

- **Reduced Lead Times and Tooling Costs:** AM eliminates the need for tooling and allows for rapid prototyping, reducing lead times. This flexibility is beneficial for developing new designs and producing small batch sizes without expensive retooling.

- **Repair and Remanufacturing:** AM, particularly DED, enables efficient repair of high-cost components, such as turbine blades, by building up material in damaged areas without requiring full part replacement.

- **Customization:** With AM, manufacturers can create customized parts tailored to specific requirements, which is valuable in applications where each component must be optimized for unique operating conditions.

All these advantages can be achieved provided that we can overcome the problems mentioned below:

- **Residual Stresses and Distortion:** The rapid heating and cooling cycles in AM cause residual stresses, which can lead to warping or cracking. Managing these stresses is critical, particularly for high-stress applications like turbine blades. Post-processing techniques, such as stress relief annealing, HIP or heat treatments, are often necessary.

- **Porosity and Defects:** AM processes can introduce porosity, microcracks, or incomplete fusion, especially in laser-based methods. These defects can

compromise the high-temperature performance of superalloys, making it crucial to control process parameters and incorporate quality control measures, such as X-ray CT scanning.

- **Powder Quality and Handling:** The quality of metal powder directly affects the quality of AM parts. Powder needs to have specific characteristics, like sphericity, size distribution, and low oxygen content, which can be challenging and expensive to achieve. Powder degradation over repeated uses can also introduce contamination.

- **Post-Processing Requirements:** AM parts often require extensive post-processing to achieve desired mechanical properties and surface finishes. Techniques like HIP improve density and reduce porosity, while machining or polishing enhances surface quality. Thermal treatments are also common to relieve stresses or improve phase distribution in the material.

- **Anisotropy:** Components produced via AM tend to exhibit anisotropic properties, meaning that strength and other mechanical properties may vary depending on the build direction. This can be addressed by optimizing the build orientation and applying specific heat treatments, but achieving isotropic properties remains a challenge.

- **High-Cost Equipment and Maintenance:** AM equipment, especially for metal powders, is capital-intensive, and the machines require controlled environments to prevent contamination and maintain powder quality. The need for regular maintenance and specialized training adds to operational costs.

4. Microstructural Control in AM of Ni-Based Superalloys

In AM, microstructure plays a crucial role in determining the mechanical performance of the final product. The following microstructural factors are carefully controlled:

- **Grain Structure:** The AM process often results in columnar grain structures aligned with the build direction, which can improve creep resistance along that direction but may reduce isotropic properties. Adjusting scanning strategies, such as rotating laser paths, can help achieve more isotropic grain structures.

Considering that the microstructure of SLM parts is influenced by small melt pools. The macro-etched image of these parts looks like fish skin. An example of this structure can be seen in Fig. 4. That is, the molten pools are placed on top of each other.

Fig. 5. contains the inverse pole figure (IPF) images showing the grain morphology of the Y-Z plane of the samples after each step of production. The as-printed sample, shown in Figure 5a, shows epitaxial grain growth in the direction of heat transfer. Such microstructure is a result of the repeated thermal cycling of the sample as subsequent layers of

powder are fused by the laser after solidification of last layer.

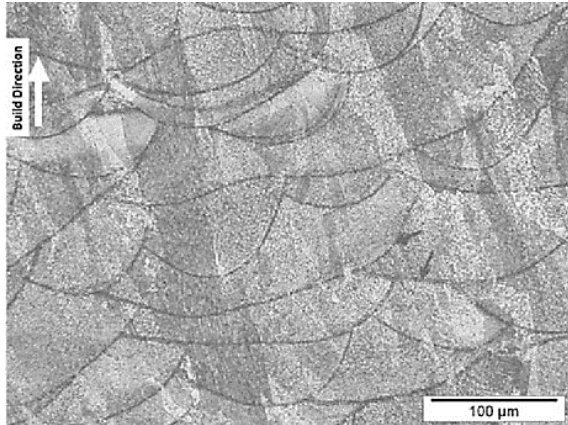


Fig. 4. The SLM as-printed microstructure showing segregation patterns of scan layers as marked by the arrows [40].

This repeated thermal cycling causes thermal gradients in the build-direction resulting in a columnar grain growth along that direction. Similar columnar grain structure has been observed during the SLM process for various materials and is a very typical feature of the SLM process [41,42]. Fig. 5.b shows the IPF after full solution. Solution heat-treatment did not have any significant effect on the grain morphology. The characteristics and the aspect ratio of the grains remained similar to the as-printed samples. Post partial solution, shown in Figure 5c, grain size increase is evident. Some grains appear to have widened perpendicular to build direction, resulting in a decrease in the aspect ratio of the grains. In fact, due to heat and pressure, the grain grows in the vertical direction to larger diameter, so that the grains have uniform size in all dimensions. This is the basis of grain growth in PM. This effect continues to occur during the 3rd and the aging, as shown in Fig. 5.d. Despite this, the post heat-treatment grain morphology still consists of grains that are elongated in the build-direction, which is known to result in anisotropic behavior [42].

Many annealing twins can also be seen in the samples microstructures. These are known to form during recrystallization in low stacking fault energy (SFE) materials like Ni-based alloys [43] and similar annealing twins have been observed to form in other Ni-based superalloys [44]. These obstruct slipping of dislocations resulting in dislocation pile-ups [42].

• **Phase Composition and Distribution:** Ni-base superalloys rely on precipitates, such as the γ' phase, for strengthening. In AM, rapid solidification can lead to non-equilibrium phase distributions, affecting mechanical properties. Specific heat treatments are applied to optimize phase composition and distribution post-build. Dastgerdi

used high speed quench to have homogeneity in microstructure after solution in his observations there was only austenite matrix and MC carbide [45].

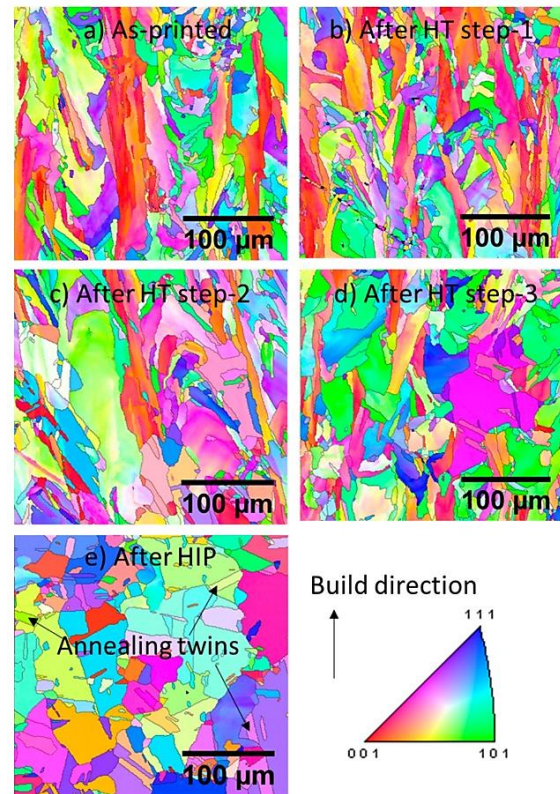


Fig. 5. Electron backscatter diffraction (EBSD) inverse pole figures of samples (a) as-printed, (b) after full solution, (c) partial solution, (d) Aging, and (e) After HIP treatment [42].

Usually, in the SLM of Ni-based superalloys, due to rapid solidification, only primary carbide and laves are seen in the matrix. Residual stress due to rapid solidification will be a strong driving force for the precipitation of secondary precipitates [35,45]. As shown TEM in the Fig. 6, in718, with a simple heat treatment, a lot of precipitates can be seen in the matrix. Normally, (γ' and γ'') precipitates require a two-stage aging with a controlled cooling rate to be synthesized [46].

Therefore, supersaturated solid solutions could be easily generated, and Nb segregated along cellular walls in the SLM-processed IN718. Herein, we regard the SLM-processed samples as being in a semi-solution state, which is different from other traditional processing techniques. Through the exploration of SLM-processed IN718 microstructures, the IN718 fabricated by SLM exhibited hierarchically heterogeneous microstructures with only a small quantity of precipitate generation. Therefore, it is crucial to explore an approach to promote the further precipitation of strengthening phase. The hierarchical submicron microstructures in the SLM-

processed samples were extremely valuable, and these properties should ideally be retained after the posttreatment processes [46].

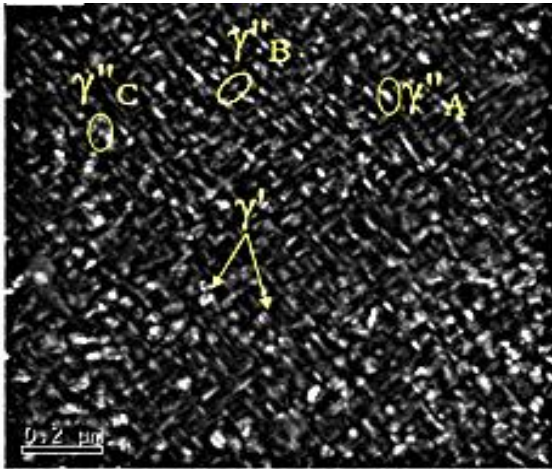


Fig. 6. Dark-field TEM images of SLM-processed IN718 after heat treatment. Dark-field image shows the morphology of precipitates [46].

Defects, such as grain boundaries, dislocations, and stacking faults, in the polycrystal can serve as favorable sites for nucleation of the intermediate phase, where the γ'' phase nucleates preferentially in IN718 alloy. As a result, the nucleation of the γ'' phase was more likely to occur along the characteristic cellular structures with lower activation energy barriers [45, 46].

Except for post-processing, the microstructure of AMed superalloys is also controlled by the parameters of these methods. Laser power and scan speed impact melt pool size, cooling rate, and layer bonding. Higher power can lead to deeper penetration but may increase the risk of residual stress and porosity, while optimized power-speed combinations improve layer adhesion and minimize defects [47]. Thin layers enhance precision but increase build time, while thicker layers may reduce detail but can improve build speed. Scan strategy (e.g., hatch spacing, rotating scan direction) affects grain orientation, residual stress distribution, and mechanical anisotropy [48, 49]. The orientation affects grain structure and mechanical anisotropy. For example, orienting the build to reduce the likelihood of stress concentrators along the critical load path can improve tensile and fatigue performance [50]. Preheating the powder bed or substrate can reduce cooling rates and residual stresses, helping to achieve more equiaxed grain structures and reduce anisotropy. Advanced sensors and cameras monitor melt pool size, temperature, and layer formation in real-time, allowing for immediate adjustments to laser parameters, reducing defects like porosity or inconsistent layer bonding. Combining laser AM with traditional manufacturing methods or post-processing directly within the AM build environment can enhance part quality. For

example, hybrid systems integrate milling with AM to improve surface finish and dimensional accuracy. The microstructure and mechanical properties of laser AM-produced superalloys are shaped by rapid solidification and build parameters, leading to unique challenges and opportunities. Key features include fine grains, columnar structures, and dendritic patterns, which influence mechanical properties like tensile strength, creep resistance, and fatigue life. To achieve optimal performance, post-processing techniques such as HIP, heat treatments, and surface finishing are essential. Advanced techniques in process control and real-time monitoring are helping to refine the microstructure further, making laser AM an increasingly viable option for producing high-performance superalloy components in demanding applications.

5. The Future of Additive Manufacturing

AM of Ni-based superalloys is increasingly used in sectors that demand complex, high performance components, including [50]:

AM is used to create turbine blades, nozzles, and heat exchangers, which require high-temperature strength and resistance to creep and fatigue. In gas turbines, AM allows for the production of components with internal cooling channels that enhance thermal efficiency. Turbine blades and combustion chambers benefit from AM's ability to create intricate cooling channels, improving efficiency and thermal management. Ni-based superalloys are sometimes used in AM-produced medical implants, particularly where high strength and corrosion resistance are essential. High-performance engines and exhaust systems use Ni-based superalloys due to their ability to withstand high temperatures and stress, and AM allows for lightweight, optimized designs.

Research is ongoing to improve AM processes and further adapt them to Ni-based superalloy production. Trends include:

New monitoring technologies, such as real-time melt pool analysis and thermal imaging, aim to improve defect detection and process control during manufacturing. AM can produce components with varying compositions across the part, optimizing properties in specific regions, like wear resistance on the surface and strength internally. Combining AM with traditional machining allows for creating components with AM's flexibility while retaining the precision and finish of conventional methods.

Efforts are ongoing to recycle metal powders and reduce the environmental impact of AM, aligning with sustainability goals in manufacturing.

6. Conclusion

AM continues to open new possibilities in designing and producing Ni-based superalloy components,

making it an invaluable tool for industries that demand high-performance materials and complex part geometries. Laser-based AM has revolutionized Ni-based superalloy production, providing unparalleled design freedom, reduced material waste, and the ability to create high-performance components for demanding applications. Despite challenges such as residual stresses, porosity, and post-processing needs, advancements in process control, hybrid manufacturing, and in-situ monitoring are helping to unlock the full potential of this technology. With ongoing research, laser-based AM is expected to play an increasingly important role in producing efficient, lightweight, and optimized components across industries.

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