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Heat treatments to improve microstructures of nickel alloys after selective laser melting

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Abstract

Selective Laser Melting (SLM) is the preferred additive manufacturing process for manufacturing high-temperature metals such as nickel alloys, and it has revolutionized the ability to create complex, previously unimaginable structures. However, the full mechanical capabilities of alloys produced via SLM cannot be achieved without methods of controlling the microstructure and addressing defects. This work reviews combinations of hot isostatic pressing and other heat treatments that can reduce porosity, anisotropy, residual stress cracking, and undesired precipitate phases and enhance overall mechanical properties. Comparative studies of post-processing treatments are documented for the Inconel 718, Inconel 625, Inconel 738, Haynes 282, and Hastelloy X alloys. Although not all nickel alloys have been well characterized, post-processing methods have consistently shown the ability to achieve many microstructural properties equivalent to (and in some cases, better than) wrought samples. In addition, many recommendations apply to all nickel alloys, and the results of these existing studies can therefore help guide future research into post-processing procedures for newer alloys.

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1. Introduction

Nickel-based alloys are widely used in applications involving extremely high-temperature and sometimes corrosive environments, such as aircraft engines, nuclear power equipment, and pollution control systems [1]. Excellent mechanical properties are derived from the precipitate phases γ’ and γ’’ that are stable at elevated temperatures. However, the presence of strengthening precipitates makes nickel alloys notoriously difficult to manufacture [2].

The introduction of additive manufacturing (AM) has great promise for industries that rely on parts with complex material performance and geometric requirements. Compared to most traditional manufacturing methods, AM improves process efficiency, reduces material waste and emissions, and allows more control over atmosphere environments [3]. As a category of AM, powder bed fusion techniques work by melting and resolidifying powdered metal feedstock with either a laser (selective laser melting, or SLM) or electron beam (electron beam melting, or EBM) in a layer-by-layer fusion process. They offer a way to fabricate fully dense metal parts in shapes that would previously be difficult or practically impossible, such as lattice structures [3, 4]. SLM in particular has been widely investigated for nickel alloys because it reduces the need to machine hardened nickel-based superalloys, can produce fine-grained structures through the rapid cooling process (which result in high strength according to the Hall-Petch relationship), and has been demonstrated on several nickel-based superalloys [5].

However, as expanded on in the following section, SLM processes can also cause several types of defects in the resulting metal. The presence of these defects has often served as a barrier to integrating SLM-produced parts into industry [3]. In response, extensive research has been dedicated to improving the microstructure of these parts. This can involve optimizing the processing parameters (e.g., modifying the metal feedstock composition, bed configuration, or laser power), using post-processing treatments (e.g., annealing or hot isostatic pressing, HIP), or a combination of the two. Recent reviews of post-processing treatments already exist for some well-studied SLM materials such as Ti-4Al-6V. This review focuses on the increasing research into nickel-based alloys. Post-processing heat treatments and HIP are specifically examined as means to improve the microstructure of nickel alloys independent of the processing parameters for the original SLM-produced part.

2. Common SLM Defects and Post-Processing Solutions for Nickel Alloys

Several mechanisms in SLM can lead to defects. Poor powder mixtures or process parameters can result in porosity from entrapped gasses [5]. The intersecting layers and tracks can cause phenomena known as molten pool boundaries, which cause elongated columnar grains and anisotropy and are common crack initiation locations under tensile stress [1, 6]. During the layer-by-layer scanning, thermally-induced residual stress builds up parallel to the scan direction and can cause distortion and cracking during post-processing or use of the part [7]. When alloys are reheated for welding or post-processing heat treatments, cracking can occur through ductility dip and strain age mechanisms when the microstructure is unable to relieve the residual stresses [8]. Alloys exceeding 4 wt% Al and Ti combined, including the Inconel 738 alloy with ~6.8 wt%, are particularly susceptible to strain age cracking while alloys such as Inconel 718 (1.5 wt%) are more resistant [5]. Regardless of the composition, the rapid cooling in the SLM process can lead to thermal stress cracking [5, 9, 10]. Finally, Nb and Mo can become micro-segregated and precipitate in the undesirable Laves phase during SLM while inhibiting the precipitation of the γ'' phase [1].

Overall, SLM-produced parts, consistent with most AM processes, generally show more microstructural defects compared to cast or wrought samples due to the localized, rapid temperature changes, buildup of residual stresses, and potential entrapment of gasses [3, 11, 12]. Research addressing porosity, anisotropy, residual stress buildup, and phase precipitation is reviewed below.

2.1 Porosity

Uncontrolled porosity can cause stress concentrations in a material that decrease its strength properties and fatigue resistance [12]. To address this, HIP is a standard post-processing treatment [5]. It closes pores and densifies a part using high pressure and temperature applied simultaneously via an inert gas, thus ensuring constant pressure from all directions and minimal deformation of the overall part geometry. The process has been demonstrated on a variety of nickel alloys. For example, HIP at 1120-1240 °C and 100-165 MPa for 3-4 h was shown to densify selective laser sintered Inconel 625 from 98.5% to 99.5% relative density [13], and a similar HIP process is used with SLM [14]. Similarly, for Hastelloy X, 1175 °C heat applied at 150 MPa for 2 h improved 99.2-99.8% dense samples to over 99.9% density, in contrast to pure heat treatment, which did not change the density [6].

However, SLM often uses the noble gas Ar as a non-reactive atmosphere, which can lead to Ar being entrapped in material and therefore limit the achievable density in HIP [12]. As the material is compressed in HIP, the elevated temperature increases the pressure inside the pores [12]. In a study with the Inconel 718 alloy, 99.985%–99.989% relative density was shown to be achieved using 1150 °C or above and 100–150 MPa, but 100% density did not occur, and Ar was detected at a 0.3 ppm concentration in the material [12]. Gonzalez et al. also found HIP applied to Inconel 625 with 99.9% as-built density did not increase density beyond 99.9% [15]. When such porosity poses a barrier to the design application, it is therefore necessary to use a vacuum environment in the SLM process rather than purely rely on post-processing [12].

2.2 Anisotropy

The anisotropy caused by SLM makes parts prone to failure when subjected to loads the weak direction. A study of Hastelloy X found that high-temperature annealing (at ambient pressure) at 1175 °C for 2 h dissolved the molten pool boundaries and resulted in isotropic properties and an increase in elongation at failure, as did a HIP method using the same temperature and timeframe with 150 MPa pressure [6]. Another study observed that heat treatment and HIP cause the anisotropic structure to partially recrystallize [16]. Similarly, for Inconel 625, research has shown that anisotropy attributed to a columnar grain structure can be reduced by applying a low-temperature solution treatment or HIP [14].

However, these high-temperature post-processing treatments cause grain growth. For example, Kreitcberg et al. found grain growth averaging up to 40-50 μm after HIP, which results in a lower tensile strength compared to the samples with no post-processing [14]. While the ductility and isotropy increased in these demonstrations, the strength tradeoff must be taken into account when determining the best heat treatment for a specific application [14, 16].

2.3 Residual stresses and associated cracking

Stress relief annealing, which consists of heating a sample to below the recrystallization temperature, has been shown to be effective at removing residual stresses in SLM-produced parts without changing the grain structure [7, 14]. An early study using steel showed 70% stress reduction after one hour of annealing at 600 or 700 °C [7]. For nickel alloys specifically, typical temperature ranges of 650-870 °C are used [14]. It should be noted, however, that other phases can precipitate at these temperatures, most notably carbide, and such phases have been observed in studies of post-processing heat treatments aimed at that range, introducing new effects on the material properties that may or may not be desirable [1, 14]. Stress relief annealing is often therefore combined with subsequent solution treatment of 1040-1200 °C, which dissolves the δ and carbide phases, both of which cause hardening [14, 17].

Heat treatment is generally effective in diminishing molten pool boundaries and restoring strength. This is shown for example by a study on Inconel 625 samples [1]. Solution treatment (1070 °C for 1 h) followed by ageing (720 °C for 18 h) increased yield strength from 576 to 813 MPa and ultimate tensile strength from 836 to 927 MPa, with a reduction in ductility from 45.2 to 43.1 percent elongation [1]. In addition, HIP was shown to restore fatigue strength after microcracking of Hastelloy X [18]. However, large, structural cracking can also occur during post-processing in the form of strain age cracking [8].

2.4 Phase precipitation

Many desirable properties of nickel alloys are from the γ’ and γ’’ precipitate phases, and conversely, the Laves phase frequently seen in as-built SLM parts causes embrittlement [1, 2]. Therefore, solution heat treatments are often used to improve the microstructure in post-processing. In a study of Inconel 625, for example, a treatment of 1070 °C was applied for 1 hour to dissolve the Laves phase, allowing the Nb to diffuse back into the matrix [1]. This was followed by a double ageing treatment in which that sample was subjected to ageing at 720 °C (for γ’’ precipitation) for 18 h [1]. This treatment was found to achieve a uniform distribution of the γ’’ phase [1]. The combination of a solution heat treatment to dissolve an unwanted phase followed by a heat treatment to participate the desirable phases is a common example of using multiple treatment steps to improve an alloy’s properties [1].

3. Treatment Procedures for Selected Alloys

In the research reviewed thus far, mechanical properties have been improved by reducing the porosity, anisotropy, residual stresses, and undesirable precipitates in as-built SLM-produced parts through post-processing heat treatments. Research has also focused directly on optimizing mechanical properties through combinations of techniques for specific alloys. Examples for Inconel 718, Inconel 625, Inconel 738, Haynes 282, and Hastelloy X are reviewed below.

3.1 Inconel 718

Inconel 718 is a common precipitate-strengthened nickel alloy for SLM, and treatments are well-studied. Several research works compare various heat treatment and HIP processes, and a review and comparison of strength, hardness, ductility and creep properties after different treatments has been presented by Hosseini and Popovich [19].

Most as-built and heat-treated samples are stronger than cast samples due to smaller grains but weaker than wrought samples due to higher porosity, although this depends on the powder processing [19]. Multiple standard treatments exist featuring different combinations of HIP (1180 C at 150 MPa for 3 h), homogenization (1065 C for 1 or 1.5 h), solution annealing (980 C for 1 h), and aging (620 C for 8 h). Heat treatments often aim for 16% γ’ and 4% γ’’ nanoscale precipitates, and HIP can close pores. However, surface finishing must also be used to improve fatigue properties, which suffer from surface defects in SLM samples [19].

3.2 Inconel 625

Inconel 625 is a solution-strengthened alloy that is nevertheless affected by precipitates, and it is also well-studied in SLM. Individual and combined effects of stress-relief, solution treatment, and high-temperature recrystallization annealing and HIP have been studied and consistently improve the microstructure and mechanical properties [11, 14, 17, 20].

In particular, stress relief post-treatment followed by recrystallization annealing can create an alloy with good ductility, nearly 100% density, and good isotropy [11, 14, 17]. A study by Zhang et al. using Inconel 625 found that stress relief heat treatments (870 °C) resulted in Laves phase precipitates growing faster than in wrought alloy by orders of magnitude. However, homogenization heat treatment (1150 °C for 1 h) could dissolve the Laves phase and produce the desired mechanical properties and microstructure [21].

Studies such as Poulin et al. show HIP can create isotropic properties, achieve yield strength and ultimate tensile strength equal to the wrought material, and increase ductility compared to the stress relief processed sample (although not to the level of the wrought sample) in tests at room temperature [11]. However, it is not always sufficient for improving fatigue life. Poulin et al. found a ~0.2 mm subsurface layer of <50-micrometer-diameter pores in SLM-produced sample linked to accelerated crack propagation compared to machined samples. Coarsening of grains during HIP promoted crack closure, but the effect was anisotropic and likely limited to low stress ratios. Therefore, like in Inconel 718, surface finishing to remove surface roughness and subsurface pores is recommended [11].

Chen et al. studied heat treatment in Inconel 625 with subsequent laser shock peening, which produced a stronger and harder alloy than either treatment alone. Heat treatment improves tensile strength by removing residual tensile stresses that can lead to cracking. Laser shock peening improves the wear and fatigue resistance by creating residual compressive stress at the surface. It uses a laser pulse to create a plasma that expands and plastically deforms the material with a high-pressure shockwave. It can reduce grain size, impede crack propagation, and help the material deform more uniformly under stress. In this application, combining heat treatment and laser shock peening changed the stress state from tension to compression and improved the microstructure through phase precipitations, apparent elimination of the molten pool boundary regions, new dislocation lines and dislocation tangles, and grain refinement [1].

Kreitcberg et al. studied stress-relief annealing (650-870 °C) alone and with recrystallization annealing (930-1040 °C), solution treatment (1040-1200 °C), or HIP (1120-1240 °C and 100-165 MPa for 3-4 h). At room temperature, ductility and isotropy increased and strength decreased with higher heat treatment compared to the as-built sample. However, at elevated temperature (760 °C), the SLM samples had comparable or greater strength to the annealed wrought alloy but consistently lower elongation at failure [14]. Further study revealed that this high-temperature embrittlement is associated with the formation of carbides at grain boundaries, and the authors recommend further research into post-processing methods to mitigate such carbides [20].

3.3 Inconel 738

While Inconel 718 and Inconel 625 are the most widely researched nickel alloys with regards to post-SLM treatments, Inconel 738 is also usable in SLM, and particular care must be taken due to its loss of ductility at elevated temperature and its susceptibility to strain age cracking and solidification cracking [8, 9].

Experiments by Wang et al. on Inconel 738 found improvements in mechanical properties from HIP and both standard and double ageing heat treatments [5]. The most effective process was HIP at 920 °C or 1175 °C for 4 h, which improved the ductility and tensile strength of the alloy, followed by double ageing heat treatment, which created a more uniform microstructure with regards to γ' precipitation and further improved mechanical properties [5]. In addition, Ding et al. conducted annealing experiments and found that stress relief annealing at 800 °C for 24 h resulted in the highest microhardness and tensile strength [22].

3.4 Haynes 282

Haynes 282 is a nickel-based alloy with both good high-temperature properties and fabricability due to its lower γ' component compared to, for example, Inconel 738. Nevertheless, it is a relatively new alloy, and there exist only two very recent studies of treatments after AM [23, 24].

Deshpande et al. found both three-stage ageing treatment and HIP could achieve γ' precipitation. The three-step heat treatment consisted of: (i) a solution treatment (2 h at 1120 °C followed by a water quench) (ii) ageing for 2 h at 1000 °C followed by furnace cooling, and (iii) ageing for 8 h at 788 °C followed by furnace cooling. Solution treatment was effective in dissolving the Laves phase although resulted in carbide precipitation. The γ' precipitate phase was identified after 950 °C heat treatment and 1000 °C treatment in that study, while a heat treatment study by Joseph et al. identified γ' only after the third step (788 °C ageing) [23, 25]. The γ' precipitation and dislocations improved hardness, yield strength, and tensile strength in subsequent measurements and decreased ductility. On the other hand, the one-step HIP maintained hardness and strength similar to the as-built samples at room temperature due to the coarsening of grains during recrystallization cancelling out the effect of stronger phases, but unlike the unprocessed alloy, the authors state that it would retain its properties at high temperature [23].

Shaikh et al. then expanded the research and produced >99.98% dense and crack-free Haynes 282 through laser powder bed fusion and the same standard 3-step heat treatment. In this study, the post-treatment alloy had γ, γ', and carbide phases comparable to a wrought sample but with more finely distributed carbide, which may reduce fatigue cracking. Tests of mechanical properties at room temperature and 800 °C found good yield strength but reduced ductility at elevated temperatures. The small grain size and anisotropy may also make the alloy susceptible to creep. Future research on failure mechanisms and recrystallization treatments for AM Haynes 282 may improve these properties [24].

3.5 Hastelloy X

Hastelloy X is commonly used in applications requiring low porosity and crack-free microstructure to accommodate high stress and cyclic loading. Research into processing parameters has shown Hastelloy X to be susceptible to cracking with certain compositions of minor alloying elements Mn and Si and in general with the rapid temperature changes that occur in SLM [26]. Crack-free SLM Hastelloy X is therefore of particular research interest.

Studies across multiple processing conditions have shown HIP to improve the porosity, heal cracks, and improve ductility and tensile strength [6, 16, 27]. Furthermore, Zhonggang et al. studied both processing parameters and post-processing solution and ageing heat treatments on samples produced with optimized parameters [28]. Heat treatments of 1000, 1100, and 1200 °C resulted in larger, less columnar grains, consistent with Etter et al., and higher temperatures were associated with more distinguished grain boundaries [28, 29]. Solution treatments above 1100 °C dissolved the carbide phases and redistributed them along grain boundaries, increasing ductility but decreasing strength, while ageing redistributed some of the carbide and re-strengthened the alloy but slightly decreased ductility [28]. Rosenthal et. al observed similar mechanical property changes with heat treatment at 1150 °C [30]. Montero-Sistiaga et al. confirmed the usefulness of high-temperature annealing by comparing heat treatments of 1177 °C and 800 °C and finding less carbide and better ductility at 1177 °C [31]. Finally, Sanchez-Mata et al. also found ductility improvements with either heat treatment or HIP, including in testing at elevated temperatures, and found that strength was comparable to a wrought alloy in either case [16].

4. Conclusions

While the SLM process is prone to defects, progress has been made in post-processing heat treatments and HIP procedures to improve the microstructure and mechanical properties of nickel-based alloys. The examples of Inconel 718, Inconel 625, Inconel 738, Haynes 282, and Hastelloy X reviewed here vary in composition, typical usage, and formability. These alloys share several common considerations, with specific recommendations varying slightly depending on the specific alloy and its failure mechanisms. Stress relief annealing, recrystallization annealing, solution treatment, ageing, combined heat treatment and laser shock peening, and HIP have been examined. These treatments are shown to improve microstructure and phase precipitations from as-built samples, and the mechanical properties can be tailored to applications.

To date, research has focused primarily on hardness, yield strength, tensile strength, ductility, and microscopic analysis to evaluate the alloy performance. While some studies have examined fatigue and creep resistance, many factors regarding long-term performance of the alloys remain to be thoroughly investigated. For example, while Poulin et al.

performed initial research into fatigue crack propagation, high-temperature tests were not included, and given the usage of nickel alloys in high-temperature applications, those investigations are important future research [11]. Additionally, while the effects of post-processing on alloys such as Inconel 718 and 625 are well-characterized, alloys such as Hayne 282 are not yet thoroughly understood [24]. Although the heat treatments and HIP considerations described here are often similar across nickel alloys and provide general guidance, there are composition-dependent differences that require future research to fully explore.

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Conflict of Interest

The author declares no conflict of interest.

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