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PROCESSING OF METALLIC MATERIALS BY SELECTIVE LASER MELTING AT ELEVATED TEMPERATURES

ZPRACOVÁNÍ KOVOVÝCH MATERIÁLŮ SELEKTIVNÍM
LASEROVÝM TAVENÍM ZA ZVÝŠENÝCH TEPLŮT

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ABSTRACT

This dissertation thesis deals with the influence of preheating on the components produced using Selective Laser Melting (SLM), also known as Laser Powder Bed Fusion (LPBF) technology. The thesis contains an overview of the current state of knowledge in the field of preheating and the physical nature of preheating. Furthermore, the work contains an overview of the effect of preheating on specific types of materials. These types of materials included in the state of the art are titanium, intermetallic, nickel and aluminium alloys, and copper. From the state of knowledge, promising research areas were identified, where preheating could lead to more efficient production using LPBF technology and to expansion of the area of processable materials. These areas include the investigation of the effect of preheating in combination with other process parameters on the residual stresses of Ti6Al4V alloy, the effect of preheating on nickel alloy Inconel 939 and copper. The premise of the Ti6Al4V and Inconel 939 topics was that preheating would reduce residual stresses, and thus will be possible to reduce the necessary amount of support structures. The results can lead to more cost-effective production using LPBF technology. This hypothesis was rejected. Despite the reduction in residual stresses in Ti6Al4V, they were not fully eliminated and, in addition, a rapid degradation of unused powder was detected, which increases production costs. The preheating of the Inconel 939, against the assumption based on behaviour of other materials, led to higher deformations and thus residual stresses, due to the evolution of precipitates. Another selected area where preheating could lead to an increase in the portfolio of processable materials is the processing of copper. Copper is a difficult to process material using LPBF technology due to its high thermal conductivity and laser reflectivity. The experiments confirmed a very positive effect of preheating on the relative density of the samples. The samples reached relative density values of over 99% when fabricated with preheating at 400 °C. Thus, preheating can significantly improve the process ability of reflective and high conductive materials. All of the results lead to a better understanding of the behaviour of the materials during processing by LPBF technology and may lead to its further expansion to more industries. The results are summarized in three publications that have been published in scientific journals.

KEYWORDS

Laser powder bed fusion, Selective laser melting, residual stress, preheating, Ti6Al4V, Inconel, copper

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ABSTRAKT

Tato disertační práce se zabývá vlivem předehřevu na výrobu komponent 3D tiskem kovů technologií Selective Laser Melting (SLM), také známou pod označením Laser Powder Bed Fusion (LPBF). V práci je obsažen přehled současného stavu poznání v oblasti realizace předehřevu a fyzikální podstaty předehřevu. Dále je v práci obsažen přehled vlivu předehřevu na konkrétní typy materiálů. Mezi tyto typy materiálů byly zařazeny titanové, intermetalické, niklové a hliníkové slitiny a měď. Z rešeršní části byly identifikovány perspektivní oblasti, které doposud nebyly dostatečně zkoumány, a kde by předehřev mohl vést k zefektivnění technologie LPBF a rozšíření oblasti zpracovatelných materiálů. Mezi tyto oblasti bylo zařazeno zkoumání vlivu předehřevu v kombinaci s dalšími procesními parametry na zbytková napětí u slitiny Ti6Al4V, vliv předehřevu na niklovou slitinu Inconel 939 a na měď. Předpokladem u Ti6Al4V a Inconelu 939 bylo, že předehřev sníží zbytková napětí a bude tak možné snížit množství podpůrných struktur během výroby, což by vedlo k zefektivnění technologie. Tato hypotéza byla zamítnuta, protože i přes snížení zbytkových napětí u Ti6Al4V nedošlo k jejich eliminaci a navíc, došlo k rychlé degradaci nepoužitého prášku, což zvyšuje náklady na výrobu. U Inconelu 939 dokonce zvýšená teplota vedla k vyšším deformacím, a tedy zbytkovým napětím v důsledku evoluce karbidické fáze. Další perspektivní oblastí, kde by předehřev mohl vést k zvýšení portfolia zpracovatelných materiálů, je měď. Měď je díky vysoké tepelné vodivosti a odrazivosti laserového záření považována za obtížně zpracovatelnou technologií LPBF. Z experimentů byl potvrzen velice pozitivní vliv předehřevu na relativní hustotu vzorků. Vzorky dosáhly hodnot relativní hustoty přes 99 % pokud byly tisknuty s předehřevem 400 °C. Bylo tedy experimentálně ověřeno, že předehřev může významně zlepšit zpracovatelnost skupiny materiálů, které mají nízkou pohltivost laserového záření a materiálů s vysokou teplenou vodivostí. Všechny výsledky vedou k lepšímu pochopení chování materiálů během zpracování technologií LPBF a mohou vést k jejímu rozšíření do dalších průmyslových odvětví. Výsledky jsou shrnuty ve třech publikacích, které byly vydány ve vědeckých časopisech.

KLÍČOVÁ SLOVA

Laser powder bed fusion, Selective laser melting, zbytková napětí, předehřev, Ti6Al4V, Inconel, měď

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1 INTRODUCTION

Additive technologies are in development for more than thirty years. In contrast to conventional technologies, where parts are produced by material removal, additively processed parts are made by gradual addition of material just where is needed. The result is a near net shape part produced without a special requirement for tooling even from the hard to process materials. Thus, those techniques open new possibilities for unique shape designs that allow fabrication of even more efficient components. Those techniques are more suitable for producing individual components with complex geometries than for serial production.

The Laser Powder Bed Fusion (LPBF) also called Selective Laser Melting (SLM) is the wide-spread technology for producing metal components. This technology uses a laser beam, which selectively layer by layer melting a metal powder. The process begins with the spread of the metal powder on the solid platform which is consequently melted by the laser beam in a specific shape according to the geometry [1]. Then the next layer of the powder is spread, and melting process continues until a complete part is created. The whole process is maintained in protective gas of argon or nitrogen. The mechanical properties of the components produced are comparable with the convention manufacturing technologies and are many times even better in static loading performance due to the specific microstructure [2, 3].

The main challenges in the LPBF process that hinder the usability in even more applications are part accuracy, requirement of support structures, high surface roughness, low fatigue mechanical properties, and low amount of proven material.

Most of the issues mentioned here are related to the residual stress originating from the nature of LPBF technology. Nonhomogeneous heating and cooling lead to high-temperature gradients and the evolution of residual stresses [4]. The residual stress causing delamination, cracks and part deformation, thus the parts must be fixed to the build platform by volumetric material or support structures. The need of support structures limits designs with inner holes and channels where support removal is impossible. The support structures also prolong the build time, increase material consumption, and postprocessing effort. Furthermore, residual stress may cause cracks which are more common in materials with poorer weldability [5]. This restricts some materials from the process ability by LPBF technology. The thermal gradient and thus residual stress can be reduced by optimal set of laser-related process parameters, scanning strategy, or preheating of the build space [6–8]. The residual stress can also be eliminated after the production run using stress relieve annealing which can reduce residual stress by more than 70% [9], however, it cannot prevent failures during component production.

This thesis focuses on the application of high-temperature preheating in LPBF technology and its influence on residual stress, relative density, microstructure, and mechanical properties of a wide variety of material. The aim is to use elevated preheating temperatures to increase accuracy by reducing residual stress and distortion. Moreover, increase manufacture efficiency by decreasing the necessary amount of support structures and increasing the material portfolio by investigating hard-to-process material.

2 STATE OF THE ART

2.1 Powder bed fusion technologies

The standard terminology for additive manufacturing ISO/ASTM 52900:2015(E) defines powder bed fusion (PBF) technologies as the process where thermal energy selectively fuses regions of a powder bed. The technologies can be divided by source of thermal energy to Laser powder bed fusion (LPBF) and Electron beam melting (EBM). The EBM uses an electron beam for powder melting. The process is maintained in vacuum and usually high-temperature preheating is applied on the powder bed. The high-temperature preheating in EBM at temperatures from 600 °C to 1100 °C is usually performed on the build surface using the electron beam that is defocused and accelerated [10, 11]. The main focus of this thesis is on the LPBF technology which takes the largest share among the PBF technologies, more than 80% compared to EBM, due to lower machine costs and precise beam resolution [12].

LPBF uses a laser beam to melt the metal powder. The process is maintained in an inert atmosphere of nitrogen or argon. LPBF can process a large amount of materials including titanium, aluminium alloys, stainless steels, and nickel-based superalloys [13]. But researchers are still focused on the introduction of the new materials which can even increase the industrial acceptance of this technology [14]. The static mechanical properties reached by the processed components are comparable to those of conventionally fabricated [13]. Nevertheless, suitable process parameters must be used to reach a fully dense part. The non-optimal process parameters lead to defects such as gas porosity when too much energy is used. On the contrary, the lack of fusion and key hole porosity can occur when insufficient energy is used in the material [15]. Another obstacle for many industries is the repeatability and reproducibility issues caused by process complexity [16]. The fusion process is mainly influenced by the setting of the laser beam, which causes a nonhomogeneous rapid local heating and cooling of the material; thus, high thermal gradients occur [4]. Large thermal gradients cause nonhomogeneous thermal expansion of the material which leads to the introduction of residual stress. The residual stress are stresses that remain in material when equilibrium with the surrounding environment is reached [4]. The residual stress is the main factor that causes in-process deformations, warping, and cracks. Thermal gradients can be reduced by preheating of the part during the production run [7]. Powder bed preheating is commonly performed using a heated base plate up to 200 °C [17], but commercial systems capable of preheating up to 500 °C and 800 °C are now also available [18].

The main benefit of high-temperature preheating in LPBF is in lower temperature gradients, thus lower residual stress [19]. Furthermore, preheating decreases the energy necessary to melt the powder [20], increases the absorptivity of the material [21], changes the wetting conditions [22–24], and increases the timescale of solidification [25]. Thus, preheating in the LBPB has the ability to increase the portfolio of proven materials, increase the component accuracy, and production efficiency.

2.2 Residual stress in the Laser powder bed fusion

Residual stresses can be divided into three groups according to the scale on which they operate (Fig. 2-1a). Type I residual stresses operate on the length throughout the part or specimen, thus on the macroscopic scale [14]. Type I residual stress is mostly discussed and studied in the literature with regard to additive manufacturing because it is responsible for global deformations of the parts when their magnitude elevates the yield strength of material. Type II residual stress is caused by a local microstructural effect, such as the thermal and elastic properties in different orientation [4]. Type II residual stresses operate on the grain scale. Type III is caused by crystallographic disorders, such as vacancies, and substitutional atoms and operates on the atomic scales. Residual stresses of type II and type III are almost always present in polycrystalline material, but are difficult to measure [26].

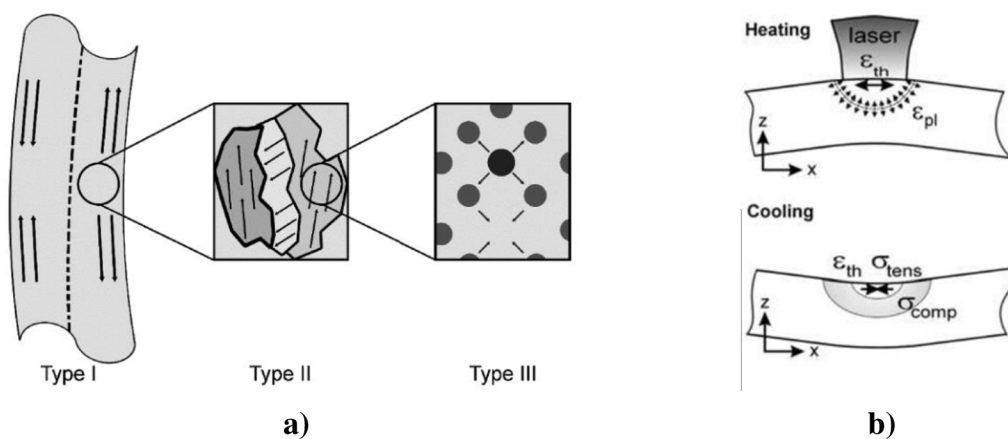


Fig. 2-1 a) Example of classification of residual stresses into three categories [14]; b) origin of the type I residual stress [27].

The macroscopic residual stresses of type I directly affect the mechanical properties and geometry of produced parts, thus they are only the type of residual stress discussed in the following text. They can also be directly affected by process conditions and laser settings. Thus, there are the most important with regards to part properties. The evolution of the type I residual stress is due to nonhomogeneous rapid heating from the laser source Fig. 2-1b. The laser source causes large thermal gradients that lead to nonhomogeneous thermal expansion of the material. The colder material beneath and around heated material restricts this expansion, which causes compression stress (Fig. 2-1b). The magnitude of compression stress may overpass the yield strength and thus plastic deformation may occur. During the cooling stage, the heated spot has a tendency to decrease its volume, which introduces tensile stress. The residual stress in the specimens is introduced in the two zones. Tensile stresses evolve on the upper and lower surfaces between the compressive stress zone in the middle of the specimen [27]. The greatest influence on residual stress has the material properties, geometry, stiffness of the base plate, scanning strategy, and temperature conditions [6–8, 28].

Residual stresses also influence the fabrication of overhangs. Overhangs and their defects were studied by Wang et al. [29, 30] who described the main challenges to their fabrication. The susceptibility to overhang defect formation is higher with increasing angle θ (Fig. 2-2). As a result of heat shrinkage, the tension forces are induced, which causes warping. The warping defect may accumulate until the unsupported part is elevated above the thickness of the layer. Then the exposed part is scanned by laser without powder and receives more energy, which can cause another deformation. In the worst case, the elevated surface may collide with the powder spreader and stop the fabrication run. Moreover, the heat dissipation of the overhang structure is limited during the fusion process because of the lower thermal conductivity of the unfused powder. The unfused powder act as an insulator with a lower thermal conductivity approximately 1-2 % of the bulk material [31]. Thus, the heat dissipation from overhang structures is lowered, which may result in large melt pool. In combination with gravity and capillarity forces, a larger melt pool may sink into supporting powder. Thus, decrease geometry accuracy and surface roughness. Therefore, the process parameters must be tuned to the lower energy input. Lower heat input can also prevent the adhesion of unfused powder particle to fused surfaces, which influences surface roughness.

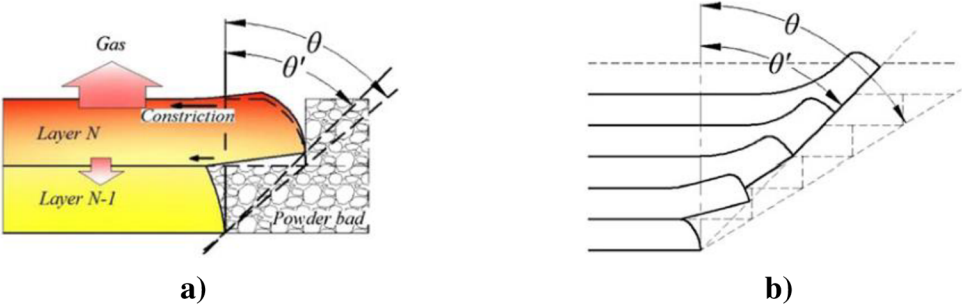


Fig. 2-2 a) warping principle; b) warping accumulation principle. [29]

Measurement of the residual stress can be divided into two main groups, nondestructive and destructive. Destructive distortion-based methods use the principle that residual stresses exist in the static equilibrium state and in any cut plane the sum of the residual stress normal to plane is zero [14]. When a new plane of the part is made, the stress must redistribute and deform the plane according to the magnitude of the residual stress. The newly deformed plane is measured and could be input into an analytical model or finite element model for residual stress calculation. The most used destructive techniques used in additive manufacturing are the hole drilling method, curvature, and contour method [32, 33]. Non-destructive diffraction techniques used in additive manufacturing are X-ray diffraction and neutron diffraction. Those techniques using Bragg's law to measure the lattice spacing and compare the results with unstrained parameters [14].

The most affecting element for the evolution of residual stress during the production run is the heat distribution in the processing part. Heat distribution can be affected by the setting of process parameters in the process that include laser-related process parameters, scanning strategy or powder bed preheating systems [34]. The powder bed preheating proved that it is an efficient technique for reducing residual stress in the process [7, 35]. The residual stresses can also be consequently eliminated after the production run by stress annealing [9], but cannot restrict the deformations and defects accrued during production. Thus, the fabricated part must be fixed on the platform directly or via support structures. Subsequently, the support structures increase the material consumption, prolong the fabrication time, and increase the postprocessing requirements.

2.3 Preheating in the Laser powder bed fusion

Preheating of the powder or part during the production run using laser powder bed fusion technology has a direct influence on the energy necessary to melt the metallic powder. The theoretical influence of the preheating can be expressed by eq. 2-1 [20]. For example, the energy necessary to melt stainless steel 316L preheated at 900 °C is 43 % lower compared to 80 °C [36].

$$Q = \rho c_p (T_l - T_0) + \rho L_f \quad (2-1)$$

where: Q (J·m⁻³) is the energy necessary for melting the powder

ρ (kg·m⁻³) - material density

c_p (J·kg⁻¹·K⁻¹) - specific heat

T_l (K) - melting temperature

T₀ (K) - initial temperature

L_f (J·K⁻¹) - latent heat

The Eq. 2-1 can be estimated that the minimum energy for melting the 316L stainless steel is $8.46 \text{ J}\cdot\text{mm}^{-3}$ using preheating at $100 \text{ }^\circ\text{C}$. Nevertheless, the commonly used energy in LPBF is around $65 \text{ J}\cdot\text{mm}^{-3}$ estimated according to eq. 2-2. The difference between these values is caused by heat loss and reflection of the laser beam under real fabrication conditions [37].

$$E_o = \frac{LP}{LV \cdot HD \cdot LT} \tag{2-2}$$

- where: E_o ($\text{J}\cdot\text{mm}^{-3}$) is volumetric energy density
- LP (W) - laser power
- LV ($\text{mm}\cdot\text{s}^{-1}$) - laser velocity
- HD (mm) - distance between two neighbouring laser paths
- LT (mm) - powder layer thickness

Absorptivity of the laser beam is an important material characteristic which is influenced by the laser wavelength (Fig. 2-3a). The commonly used infrared lasers with a wavelength of around 1064 nm have good absorptivity on steel and nickel alloys, but, for example, absorptivity on copper, gold, and aluminium is less than 10% on the smooth surfaces. For that purpose green and blue lasers are now under development [38]. However, preheating has a decreasing effect on material reflectivity (Fig. 2-3b). Thus, it is a potential method that leads to the processing of a reflective material with infrared lasers.

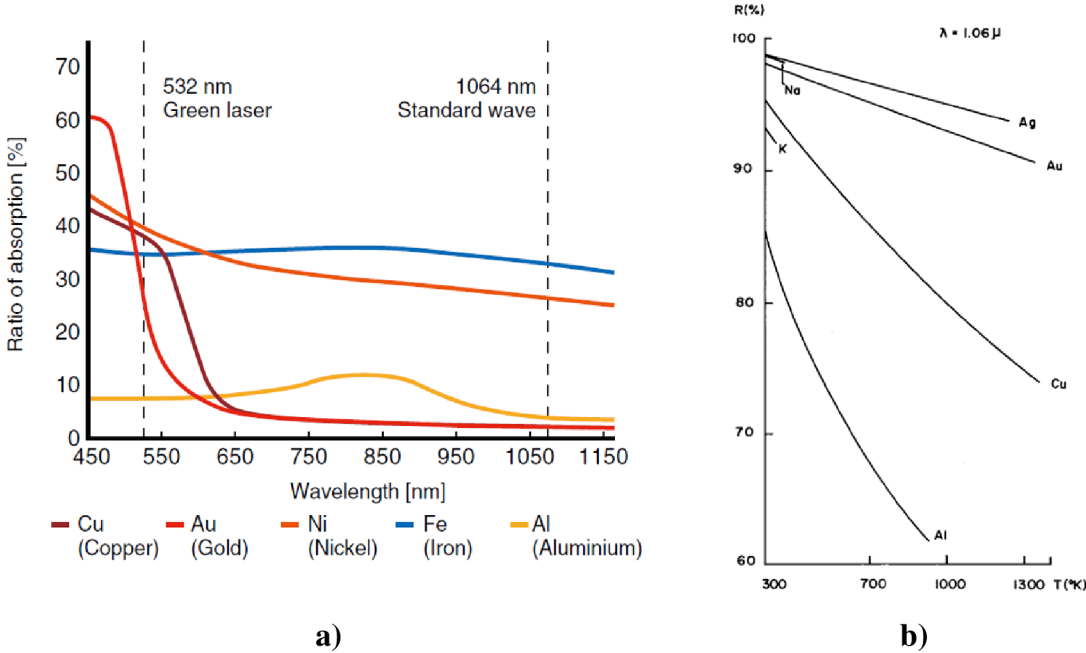


Fig. 2-3 a) influence of the absorption ratio on the laser wavelength [39]; b) influence of the temperature on the material reflectivity for 1064 nm wavelength [21].

Powder bed preheating in LPBF technology is in most cases realized using a preheated base plate [7, 40]. For preheating, resistive heating elements or induction coils are used. The preheating of the base plate has a disadvantage when high production runs are realized. The temperature of the top surface of the part gradually decreases during fabrication; thus, a non-consistent condition and differences in the microstructure were detected [41]. Thus, a different preheating system was developed and tested. The preheated base plate preheats the fabricated part and usually the new spread powder layer has a much lower temperature. Thus, the powder feedstock preheating device can decrease the temperature differences between the new and the already deposited powder layer [42]. The powder is preheated before it is spread on the manufactured part. For even more homogeneous conditions, the inertial atmosphere preheating device can be used [43]. The highest preheating temperatures were reached using an additional laser system which preheats the powder of the newly recoated layer before the laser scan the part. The preheat temperature in those cases can reach 2500 °C, which is suitable for the fabricating of ceramic materials without inner defects [44, 45]. The most efficient solution for homogeneous preheating is the combination of different types of preheating systems [46]. However, each material needs its individual preheating conditions to achieve an optimal result [47].

2.4 Influence of preheating on the selected materials processed using LPBF

The following text is divided into material groups for better readability. The aim is to define the influence of in-situ preheating on the properties of the fabricated material and to observe the suitable temperature ranges for group of materials.

2.4.1 Titanium alloys

The titanium alloys offer an exceptional strength to weight ratio, therefore, are highly desired for complex parts fabricated using LPBF. The most used titanium alloy is Ti6Al4V. The predominant microstructure of Ti6Al4V observed in LPBF is composed of columnar grains and fine martensitic needles [48]. This microstructure leads to low ductility which may lead to in-process crack formation and warping due to high residual stresses. The martensitic evolution is caused by a rapid cooling rate of more than 410 K/s needles [48]. However, the ductility can be increased after production run by heat treatment. However, in-process defects must be minimized, for example, by process parameter adjustment or preheating [7, 49].

For in-situ martensitic decomposition it is necessary to acquire a temperature higher than 600 °C for a sufficient time [49]. This can be achieved by a shorter time between scanning adjacent layers, a higher layer thickness, lower density of support structures, and a larger specimen size [49]. However, those techniques are not always reachable while producing real parts. For example, part size cannot be changed, the scanning time depends on part size, thus cannot be lowered, a higher layer thickness would increase surface roughness and a lower support structures density may lead to warping. Thus, preheating may be a proper technique that is also applicable to real part production.

It was found, that preheating at 570 °C leads to α' martensitic decomposition to $\alpha+\beta$ structure which led to the increased of yield strength to 1176 MPa and elongation by 66 % against preheating at 100 °C [7]. Furthermore, the preheating temperature higher than 570 °C led to a decrease of residual stress to zero. The residual stresses can be further decreased using the meander scanning strategy with a rotation angle of 90° [6]. Furthermore, the remelting scanning strategy can be used to reduce residual stress. However, it may have a negative impact on mechanical properties and porosity [6]. Residual stresses can also be influenced by laser-related process parameters. The longer exposition time (lower laser velocity) and lower laser power with persisted energy density lead to lower residual stresses [28].

At present, studies devoted to the fabrication of titanium alloys using LPBF have been investigated the effect of preheating on the residual stresses, on static mechanical properties, and on the microstructure. Studies were mainly focused on the effect of preheating without tuning other process parameters. For example, Ali et al. [7] did not tune laser-related process parameters and, as they found, it has a negative impact on the mechanical properties while high temperatures were used. Therefore, a study that would assess the multifactorial experiment with a combination of process parameters and preheating is missing.

2.4.2 Nickel-based alloys

Nickel-based alloys are susceptible to crack formation with an increasing content of Ti and Al elements [50]. Preheating was presented as an efficient method for processing nickel-based alloys, which are susceptible to cracking, using LPBF. However, extremely high preheating temperatures up to 1200 °C were used [51].

In addition, preheating can decrease the residual stresses. Preheating up to 150 °C of IN718 decreased σ_z (building direction) residual stresses by 90 MPa compared to 50 °C [52]. The σ_x residual stresses did not significantly change in this temperature range. However, preheating had an effect on the microstructure, where with increasing preheating temperature, the inner-dendritic spacing and width of precipitates also increased. It may explain the slightly increased hardness values that increased from 310.8±8.9HV to 314.9±11.3 HV when the temperature changed from 50 to 100 °C. The development of residual stress of IN718 according to the preheating temperature was simulated up to the preheating of 500 °C [53]. The analytical model showed that up to 200 °C the residual stresses will decrease. This was also experimentally confirmed. However, according to the prediction, higher preheating temperatures would lead to increased residual stresses due to heat accumulation and grain coarsening. This prediction was not tested experimentally.

2.4.3 Copper

Copper exhibits excellent thermal and electrical conductivity thus, is a desired material for heat exchangers, electric applications, and other applications [54]. However, the fabrication by LPBF brings issues related to the low laser energy absorptivity of lasers with wavelength 1000-1100 nm, high thermal conductivity, and oxygen affinity [55–58]. These issues researchers tried to overcome by using high-powerful infrared lasers with power up to 1 kW, alloying or adding element into pure copper, using preheating, and by using lasers with wavelength with better absorptivity [59]. However, even high-power lasers or blue diode lasers did not lead to fully homogeneous specimens [60, 61]. The researchers mainly focused on the adjustment of the laser-related process parameters and only one study was performed on the use of preheating at 100 °C [62]. Preheating has the ability to decrease laser power delivered from the laser source [20, 40], decrease the high thermal conductivity of copper [63], and decrease the reflectivity of the laser [21]. These factors can lead to copper processing with high relative density.

2.5 Analysis and conclusion of literature review

Based on analysis of the literature review, perspective research topics were pointed out for increasing knowledge of processing materials using LPBF. The first topic aims to process titanium Ti6Al4V and nickel-based IN939 alloys using high-temperature preheating. The review of the literature showed that there are still unknown gaps in knowledge of how Ti6Al4V behaves in high-temperature preheating in combination with other process parameters. Furthermore, the behaviour of IN939 under high-temperature preheating was simulated, but it needs to be experimentally tested. Those studies will further increase the knowledge about how process parameters and their combination affect residual stresses and relative density. The assumption is that higher preheating will lead to reduction of residual stress, which can be further used to reduce the quantity of necessary support structures. Thus, increase productivity and decrease financial expenses of LPBF by reducing manufacture time, waste material, and easier postprocessing.

The second perspective topic aims at increasing material portfolio. Preheating in LPBF has the ability to increase relative density. Moreover, it can act as another heat source, which can decrease laser power requirements. Thus, it can significantly improve the relative density of copper processed by using LPBF equipped with infrared lasers. It can lead to the fabrication of complex parts with unique properties.

3 AIMS OF THE THESIS

This dissertation thesis aims to experimentally investigate the effect of preheating on the Ti6Al4V alloy, Inconel 939 nickel-based alloy, and copper processed using LPBF. The main focus will be on the reduction of residual stress in the case of Ti6Al4V and IN939. Copper research will focus on clarifying the preheating effect in combination with other process parameters to maximize relative density. To achieve the main goal of this thesis, the solution of following subgoals will be necessary:

- Identification of main process parameters which affect the residual stress
- Finding a suitable method for residual stress measurement
- Finding a proper method for assessing multivariable experiments
- Elucidation of the effect of preheating and other process parameters on the residual stresses and relative density of Ti6Al4V specimens
- Elucidation of the effect of preheating on the residual stresses and relative density of IN939 specimens
- Defining the effect of high-temperature preheating and other process parameters on the relative density of copper specimens processed using infrared fiber laser

3.1 Scientific questions

Q1. Can the right combination of process parameters and preheating to 550 °C lead to the elimination of residual stresses in Ti6Al4V alloy and the cost-effective production of components manufactured using LPBF?

Q2. How does preheating affect the residual stresses of the nickel-based alloy IN939 processed using LPBF?

Q3. What is the effect of high-temperature preheating on the relative density of copper samples prepared with an LPBF system using infrared fibre laser?

3.2 Hypotheses

H1. Preheating temperature can significantly reduce residual stresses in Ti6Al4V [8]. However, residual stresses are also influenced by other process parameters, such as laser velocity, laser power, dwell time, and support structures [28, 51]. Thus, a proper combination of preheating and other process parameters should lead to the elimination of residual stresses even with preheating temperatures lower than 570 °C. The temperature of 570 °C was stated as the level for residual stress elimination [8].

H2. The effect of preheating was evaluated on different types of material and it was concluded that each group of materials needs its own process conditions [47]. The situation with preheating is the same; the appropriate magnitude of this parameter should be found. In general, a reduction in residual stresses can be expected at higher preheating temperatures [8]. Thus, residual stresses should decrease even in the IN939 alloy. However, excessive preheating can even increase residual stresses, as was simulated for the IN718 alloy [69].

H3. High reflectivity and thermal conductivity restrict the process ability of copper using LPBF [80]. Thus, the high-powerful laser systems must be used, or new lasers with different wavelengths must be developed [38, 86, 88]. High-temperature preheating has the ability to reduce those negative effects [84, 85]. Moreover, with applied preheating less energy must be acquired by the laser [20, 40]. Thus, those factors have potential to overcome issues with processing copper using infrared fiber lasers, and it can lead to homogeneous production of copper components.

3.3 Thesis layout

The dissertation thesis is composed of three scientific papers published in peer-review journals with an impact factor. The first article *Effect of Process Parameters and High-Temperature Preheating on Residual Stress and Relative Density of Ti6Al4V Processed by Selective Laser Melting* [I.] aims to answer the first scientific question of what is the proper combination of process parameters and preheating to reach the lowest residual stresses of the Ti6Al4V. The observed parameters were hatch laser velocity, hatch laser, power, border laser velocity, dwell time, and preheating up to 550 °C. Furthermore, the article discusses the effect of preheating on the unfused powder and its reusability. The second article *Effect of Preheating on the Residual Stress and Material Properties of Inconel 939 Processed by Laser Powder Bed Fusion* [II.] answers the second scientific question on how preheating affects the residual stresses of IN939. The effect of preheating on residual stress was experimentally tested up to preheating at 400 °C. Furthermore, the mechanical properties, macrostructure and microstructure changes, and also powder reusability were discussed. The third article *Effect of high-temperature preheating on pure copper thick-walled samples processed by laser powder bed fusion* [III.] aims to elucidate the effect of high-temperature preheating on copper, which is the subject of the third scientific question. In that article, a multivariable experiment focused on maximization of the relative density of the copper wall specimens was tested. The proper combination of laser power, laser velocity, layer thickness, hatch distance, sample thickness, and preheating up to 400 °C leading to maximum relative density was evaluated.

- I. MALÝ, Martin, Christian HÖLLER, Mateusz SKALON, Benjamin MEIER, Daniel KOUTNÝ, Rudolf PICHLER, Christof SOMMITSCH a David PALOUŠEK. Effect of Process Parameters and High-Temperature Preheating on Residual Stress and Relative Density of Ti6Al4V Processed by Selective Laser Melting. *Materials* [online]. 2019, **12**(6), 930. ISSN 1996-1944. Available at: doi:10.3390/ma12060930



Journal impact factor = 3.748, Quartile Q1 (Metallurgy & Metallurgical Engineering)

Citations: 48

Author's contribution: 70%

- II. MALÝ, Martin, Klára NOPOVÁ, Lenka KLAČURKOVÁ, Ondřej ADAM, Libor PANTĚLEJEV a Daniel KOUTNÝ. Effect of Preheating on the Residual Stress and Material Properties of Inconel 939 Processed by Laser Powder Bed Fusion. *Materials* [online]. 2022, **15**(18), 6360. ISSN 1996-1944. Available at: doi:10.3390/ma15186360

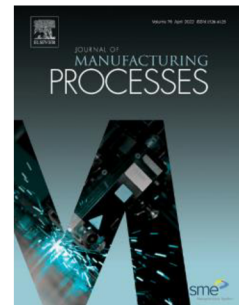


Journal impact factor = 3.748, Quartile Q1 (Metallurgy & Metallurgical Engineering)

Citations: 0

Author's contribution: 33%

- III. MALÝ, Martin, Daniel KOUTNÝ, Libor PANTĚLEJEV, Laurent PAMBAGUIAN a David PALOUŠEK. Effect of high-temperature preheating on pure copper thick-walled samples processed by laser powder bed fusion. *Journal of Manufacturing Processes* [online]. 2022, **73**, 924–938. ISSN 15266125. Available at: doi:10.1016/j.jmapro.2021.11.035



Journal impact factor = 5.684, Quartile Q2 (Engineering, Manufacturing)

Citations: 7

Author's contribution: 52%

4 MATERIAL AND METHODS

4.1 Specimens fabrication

All specimens were fabricated on the SLM 280HL 3D printer equipped with 400 W ytterbium fibre laser YLR-400-WC-Y11 (IPG Photonics, Oxford, USA) with a focus diameter of 82 μm and a Gaussian shape power distribution. As a protective gas, argon was used for Ti6Al4V and IN939, and nitrogen for copper. The powder humidity was measured before each experiment using a Hytelog hydrothermometer.

For powder bed preheating in study I., focussed on Ti6Al4V alloy, a heating platform from SLM Solutions Group was used. The device was able to preheat the build platform up to 550 $^{\circ}\text{C}$, but the build area was reduced to a cylindrical shape with 90 mm in diameter and 100 mm in height. For preheating, a resistive heating element was used and the temperature was controlled by a thermocouple placed below the base plate made of stainless steel. The powder bed preheating in studies II. and III. was realized by an in-house manufactured heating device. The resistive heating elements in this heating device can preheat a top surface of the building platform up to 400 ± 10 $^{\circ}\text{C}$. The temperature was controlled by a PID regulator and by a thermocouple placed below the base plate. The device was calibrated before the start of the printing process by measuring the build surface temperature and its dependence on the set temperature on the PID regulator. The samples were printed on a base plate made of stainless steel in the II. study and copper in the III. study.

4.2 Powder characterization

The chemical composition, particle shape, and size distribution characteristics of the powders used for fabrication of the specimens were measured. The powder shape was evaluated by SEM. The chemical composition was determined by EDX and particle size by the laser diffraction method. The powders were fabricated by the gas atomization method.

In study [I.], Ti6Al4V powder was used with a particle mean size of 43 μm and a median size of 40.9 μm . Particles up to 29.97 μm represented 10% of particle distribution, while particles up to 58.61 μm represented 90%. The chemical composition acquired by EDX fits to the standard composition of the Ti6Al4V (Tab. 4-1).

Tab. 4-1 Chemical composition of virgin Ti6Al4V powder in weight percentage (wt. %).

Al	C	Fe	V	O	N	H	Ti
6.38	0.006	0.161	3.96	0.087	0.008	0.002	Bal.

In the study [II.], IN939 powder was used with a particle mean size of 35.8 μm and a median size of 34.3 μm . Particles up to 24.7 μm represented 10% of the particle distribution, while particles up to 48.7 μm represented 90%. The chemical composition acquired by EDX detected values of IN939 powder composition (Tab. 4-2).

Tab. 4-2 Chemical composition of virgin IN939 powder in weight percentage (wt. %).

Cr	Co	Ti	W	Al	Ta	Nb	Ni
20.00 \pm 0.40	18.23 \pm 0.90	3.83 \pm 0.45	1.83 \pm 0.15	1.63 \pm 0.15	1.60 \pm 0.10	0.83 \pm 0.15	51.57 \pm 0.75

In the study [III.], copper powder of 99.5% purity was used with a particle mean size of 25.9 μm and a median size of 25.7 μm . Particles up to 22.8 μm represented 10% of particle distribution, while particles up to 29.4 μm represented 90%. The chemical composition of copper powder, declared by material supplier, is stated in Tab. 4-3.

Tab. 4-3 Chemical composition of virgin copper powder delivered by vendor.

Cu (wt %)	As (ppm)	Bi (ppm)	S (ppm)	Sb (ppm)	Ag (ppm)	Mg (ppm)	P (ppm)
Bal.	<20	<20	<20	<15	<10	<10	<10
Pb (ppm)	Si (ppm)	Sn (ppm)	Zn (ppm)	Al (ppm)	Cd (ppm)	Co (ppm)	Cr (ppm)
<10	<10	<10	<10	<5	<5	<5	<5
Fe (ppm)	Mn (ppm)	Ni (ppm)					
<5	<5	<5					

4.3 Measurements methods

Measurement methods are described in detail in the attached studies. In this chapter, methods for residual stress and relative density evaluation are described.

4.3.1 Residual stress measurement

Residual stress was indicated by deformations of specially designed bridge-like specimens Fig. 4-1. The bridge curvature method is often used in LPBF for fast comparison of process parameters and their influence on residual stress [66, 67]. The distortions were measured on the top surface in half-cut state using the 3D scanner Atos TripleScan 8M.

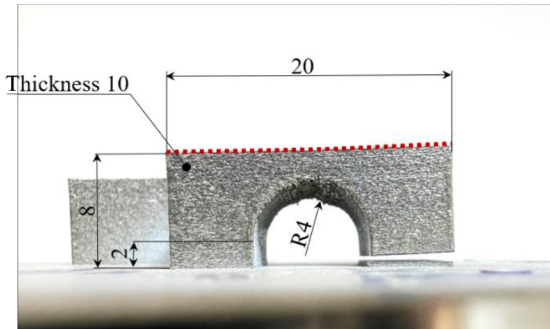


Fig. 4-1 Bridge-shaped specimen in the cut state with dimensions and marked top surface (red dotted line).

4.3.2 Relative density measurement

Relative density was measured in all publications by optical method from the polished cross-sectioned specimens. The relative density was evaluated in ImageJ v. 1.52k software in the inner area of the cross-sectioned specimens. The images for evaluation were converted to 8-bit type and relative density was evaluated with an automatic threshold.

4.4 Experimental design

In the experiments, a combination of process parameters was usually tested. Therefore, to determine their influence on the observed result, the methods for experimental planning were used. The surface response design and fractional factorial methods were also used to decrease possible combinations of process parameters. Therefore, decrease the number of specimens. The acquired data were analysed in Minitab software. The variables tested with the results are well described in the attached studies.

5 RESULTS AND DISCUSSION

In the experimental study [I.] *Effect of Process Parameters and High-Temperature Preheating on Residual Stress and Relative Density of Ti6Al4V Processed by Selective Laser Melting*, the influence of various process parameters in combination with preheating on residual stress and relative density of Ti6Al4V was tested. Process parameters that were experimentally tested were selected according to the literature review. The tested process parameters were: hatch laser speed, hatch laser power border laser velocity, waiting time between adjacent layers, and powder bed preheating up to 550 °C. For a comprehensive evaluation, the surface response design method was used to find the process parameters that have the greatest influence on residual stress and relative density. Furthermore, the evaluation method has found their optimal combination. At the end of the study, the effect of preheating on the unfused powder was evaluated.

The range of laser-related process parameters was defined according to standard process parameters used for Ti6Al4V adjusted for preheating temperature of 200 °C. The tested preheating temperature range was from 200 to 550 °C, which was the maximum of the used preheating device. The hatch laser power was tested in range of 100 to 275 W, hatch laser velocity from 700 to 1100 mm/s, and border laser velocity from 350 to 800 mm/s. Overall, preposition was that with higher preheating temperature, it will be possible to reach fully homogeneous specimens with higher laser velocities and thus, higher production rate. The delay time (time between scanning adjacent layers) was tested in the range of 22 to 73 s.

The distortions of the specimens and thus the residual stresses were mainly affected by preheating. The analysis of variance also showed that preheating and hatch laser power have a linear characteristic on the distortions, however, the hatch laser velocity and dwell time have a non-linear characteristic. The lowest distortions can be reached according to the regression model with the highest laser power, the lowest laser velocity, the shortest dwell time, and maximum preheating. The linear contribution of preheating was calculated as 46.31%. The second most influencing parameter was hatch laser power with a contribution of 17.22%. The increasing dwell time led to higher distortions and the parameter had 5.26% linear contribution effect on the distortions. Thus, the results showed that the highest energy density in combination with the highest preheating and the shortest dwell time leads to the lowest distortions. This means that the highest temperature accumulated in the specimens during the production run can be expected. This conclusion fits the overall observed behaviour of Ti6Al4V [8, 51] and can be explained as the slowed cooling speed and longer time in the ductile state which led to the relieve of internal stresses [52]. The overall distortions decreased, but they were not fully eliminated and the specimen without distortion was not fabricated. The results show that a higher energy density can lead to even lower distortions in combination with a preheating of 550 °C. However, slow laser velocity can significantly prolong fabrication time, and high laser power can lead to increased porosity [70].

The result showed that the relative density of the Ti6Al4V specimens was mainly influenced by hatch laser power and velocity. These parameters had together a linear contribution of 84.96% to the relative density. The dwell time in the observed range had a minimum linear contribution of 1.58% to the relative density. The preheating temperature had a positive effect on relative density. The linear contribution was 2.88%. Thus, it was confirmed that preheating has a positive effect on the relative density. However, in the case of Ti6Al4V, the contribution of preheating in the range of 200 to 550 °C on the relative density was less than 3%, but it can be important for other materials, for example copper. Furthermore, higher laser velocities can be used in combination with preheating. Thus, it has a potential to increase productivity.

The important conclusion was made with the effect of preheating on the unfused powder. High-temperature preheating led to visible changes in the colour of the Ti6Al4V powder. Thus, the unfused powder was analysed, and the source of changed optical properties was discovered. Preheating at 550 °C led to rapid increase in oxygen and hydrogen content, which exceeded the maximum values allowed by the ASTM B348 Grade 5 standard. The higher temperature increased the diffusion rate of oxygen and hydrogen [91], which are present due to the residual oxygen content in the inert atmosphere and the moisture contained in the powder. Oxygen content in Ti6Al4V increases ultimate and yield strength, but decreases ductility [92–94]. The ductility is also decreased by hydrogen, which can lead to part failures; thus, the content of hydrogen and oxygen is strictly monitored [95, 96]. The content of oxygen and hydrogen was not measured in the produced parts. However, powders that contain an elevated amount of these elements are not reusable for critical components because of the risk of failure. Standardly used preheating at 200 °C did not cause a change in hydrogen content, but a slightly increased oxygen content was detected. However, the chemical composition of the powder used in combination with 200 °C preheating did not exceed the maximum allowed value. Thus, it is still recyclable.

The first study showed that preheating at 550 °C of Ti6Al4V in combination with the process parameters can lead to the significant reduction of residual stress. However, the residual stresses were not fully eliminated. Furthermore, the increased oxygen and hydrogen content in the unfused powder restricts its reusability. Thus, this method is not cost effective in combination with relatively expensive titanium powder. The solution may be in using vacuum instead of an inert atmosphere and also in minimizing the moisture in the powder. However, the vacuum system will further increase the machine and operating costs. Further research was conducted with IN939, which is the heat resistant alloy; thus, the problem with oxygen content may be minimal.

The second study *Effect of Preheating on the Residual Stress and Material Properties of Inconel 939 Processed by Laser Powder Bed Fusion* [II.] aimed mainly at evaluating the preheating on residual stresses in the IN939 alloy. Furthermore, the effect on relative density, macrostructure, microstructure, mechanical properties, and unfused powder was observed. In this study, the effect of preheating was evaluated at the three levels; room temperature, 200 °C, and 400 °C.

The results of the deformation of the bridge curvature specimens showed that due to the preheating of 400 °C, the distortions and therefore the residual stresses unexpectedly increased. This result contradicts studies of other materials, such as aluminum, steels, and titanium [8, 35, 40] and the conclusion of the first study [I.] with Ti6Al4V. Furthermore, studies on IN718, which is a nickel-based alloy, showed that preheating from room temperature to 200 °C should decrease residual stresses [68, 69]. However, the IN718 simulation showed that higher preheating temperatures can even increase residual stresses due to overheating and grain coarsening [69].

IN939 showed a minor increase in distortion of 4.2 % in bridge curvature specimens when the preheating temperature changed from room temperature to 200 °C. A higher increase in the distortion was observed when preheating was used at 400 °C. Then the distortions increased about 16.2 % compared to room temperature.

The preheating of the base plate had an influence on the mechanical properties of IN939. The mean hardness and tensile properties such as ultimate tensile strength and 0.2 % proof stress increased and the elongation at break decreased. The specimen fabricated at room temperature had a mean hardness of 321 ± 26 HV 1 and the specimen at 200 °C 326 ± 19 HV 1. The hardness increased significantly to 359 ± 22.5 HV 1 when 400 °C preheating was applied. A similar trend was observed with the tensile properties. When room temperature and 200 °C preheating was applied, there was just a minor influence on the ultimate tensile strength, 0.2 % proof stress, and elongation at break. However, the ultimate tensile strength and 0.2 % proof stress increased by 26 % and 25 %, respectively, and the elongation at break decreased by 48.8 % when comparing the room temperature and the preheating to 400 °C.

The relative density of IN939 specimens did not change significantly and in all preheating levels was higher than 99.8%. No cracks were observed in any specimen. The difference in macrostructure was observed in the mean width of the melt pool. The mean melt pool width of the room temperature specimen was measured as 88.5 μm, 101.7 μm and 123.4 μm for the specimens preheated at 200 °C and 400 °C. Thus, melt pool width increased by 39.4% compared room temperature and 400 °C means a lower cooling rate can be expected.

The lower cooling rate and thermal gradients caused by preheating had a significant effect on the microstructure of IN939. Preheating at 400 °C resulted in wider columnar grains by 16% compared to room temperature. However, the overall frequency of occurrence of columnar grains decreased. The decrease in their occurrence is attributed to the reduced thermal gradients, which was also detected in the IN718 study. Preheating affects the orientation of the fine grains and contributes to the columnar to equiaxial transition by reducing the thermal gradients [68, 97]. Furthermore, the carbide phase was detected in specimens made of IN939.

Excellent mechanical properties at elevated operating temperatures of IN939 are usually acquired by precipitation hardening heat treatment. Heat treatment leads to precipitation of γ' phase and carbides. The precipitates detected by backscattered electron SEM are composed of MC-type carbides based on their location between grains, contrast, and size. The preheating temperature had a significant influence on the size and appearance of the carbide phase. The mean size of the carbide phase increased from 0.061 μm at room temperature by 41% at preheating at 200 °C and by 72% at preheating at 400 °C. The occurrence of carbide phase slightly decreased at preheating to 200 °C by 8.7% compared to the value of the room temperature specimen where $1.26 \pm 0.41 \mu\text{m}^{-2}$ was measured. However, preheating at 400 °C increased the occurrence of carbide phase per area by 23.8% compared to room temperature.

In addition, a significant influence of location on the specimen was detected. The three locations were measured, bottom, center, and top of the specimens. The location at the top is approximately in distance of 7 mm from the bottom location. The size and occurrence per area of the carbide phase gradually dropped from the bottom to the top location. The most significant drop occurred in specimens preheated at 400 °C. Then the average grain size and occurrence per area dropped by 14.6% and 37.8% compared the bottom and upper locations of the specimens.

The results of occurrence per area and size of the carbide phase showed that higher preheating temperature leads to increased concentration and size of carbides in IN939 alloy. Furthermore, a significant effect on the size and concentration of the carbide phase had time for which the specimens were exposed to elevated temperatures. The size and amount of carbide phase correspond to the measured tensile properties, hardness, and deformations. The carbide phase increased the ultimate tensile strength, 0.2 % proof stress, and hardness, but decreased elongation at break. Moreover, formation of precipitates caused the increased level of inner residual stresses and higher deformation of the specimens.

The chemical composition of the main alloy elements in the unfused powder detected by EDX was not changed. Furthermore, the powder grains of the powder used during preheating at 400 °C were not sintered together. However, the EDX analysis, which compared the virgin and powder used at preheating of 400 °C, detected a slightly increased oxygen content, but the concentration did not exceed the detection limit of 2 wt. % of XRD analysis. The oxygen content increased even though the concentration was strictly controlled and kept below 0.1 %.

The study of IN939 and its behaviour under high-temperature preheating showed that an increase in the preheating temperature cannot automatically acquire lower residual stresses. Although the unfused powder did not rapidly oxidise using the preheating temperature of 400 °C, the residual stresses increased due to the evolution of the carbide phase. Thus, preheating as a method for residual stress reduction is not appropriate for IN939.

The third study *Effect of high-temperature preheating on pure copper thick-walled samples processed by laser powder bed fusion* [III.] aimed to elucidate the effect of preheating 400 °C and other process parameters on the copper specimens fabricated using LPBF. The first study showed that preheating had the positive effect on the relative density of Ti6Al4V specimens. Thus, the preposition was that it will also have a positive impact on the relative density of copper. Therefore, the main objective of the third study was to fabricate homogeneous thin-walled specimens. Moreover, the effects of layer thickness, laser speed, laser velocity, hatch distance, sample width, scanning, and remelting strategy were studied. The copper material is suitable for its high thermal conductivity for the fabrication of coolers that are mainly composed of thin wall structures. Thus, the objective was to fabricate walls with thicknesses of 0.5 to 1.7 mm. The multivariable experiments were evaluated by surface response design and fractional factorial method.

The copper is a relatively new material in LPBF therefore, the process parameters leading to the maximum relative density are unknown. Thus, the correct combination needed to be set. For that purpose, the experiment that evaluated laser power, laser velocity, hatch distance, and sample width was conducted. The 31 specimens were fabricated at preheating of 200 °C and once with a layer thickness of 0.05, and second with 0.03 mm. The maximum value of the relative density of samples with 0.05 mm layer thickness was 88.9% and samples with 0.03 mm 95.9%. With decreasing layer thickness, the mean relative density increased by 7%. Thus, the layer thickness was identified as the most influencing parameter for the relative density. According to this experiment, the combination of laser-related process parameters that lead to maximum relative density was found as: laser power 400 W, laser velocity 505 mm/s.

The effect of preheating 400 °C was tested on specimens which process parameters were set for the fractional factorial evaluation method. Preheating at 400 °C showed a significant improvement in relative density that increased in mean value by 4.4% compared to 200 °C specimens. The relative density measured by optical microscopy of some specimens was greater than 99%. The main reason was attributed to a much smoother build surface, which is one of the conditions to reach a high relative density [75, 98]. The smoother surface was attributed to the wider weld tracks and improved wetting conditions that were observed in the thin-wall experiment. Preheating decreased the cooling rate, therefore the melt pool had more time to spread. However, in all specimens, unconnected layers were observed, which can decrease thermal and mechanical properties [99]. Copper has a high affinity to oxygen and the creation of a thin oxide film that hinders wettability and flowability [78]. Thus, the issue with unconnected layers was attributed to oxide layers. This conclusion was confirmed by EDX element mapping, which confirmed oxides in voids between unconnected areas. However, preheating at 400 °C led to oxidation and sintering of the unfused powder. The SEM and EDX analysis confirmed a high level of oxidation of the powder grains. After the production run where preheating was used at 400 °C, the powder grains were covered with oxide layers with a thickness of 1 to 3 µm. However, the increased oxidation of the copper specimens was not detected.

Remelting in combination with 400 °C preheating was tested to further increase relative density. Remelting had a positive impact on the relative density, which in mean value increased by 2.8%. Remelting further increased the temperature in the bulk material, which increased the time required for dissolving oxides. Oxides are less stable at elevated temperatures [78].

This study showed that copper can be processed by a relatively low powerful laser with a maximum laser power of 400 W and wavelength of 1064 nm and reach a relative density over 99%. However, the high relative density was reached just in combination with preheating at 400 °C. Nevertheless, the main issue was identified in oxidation, which hinders bonding of layers and tracks. Preheating in combination with remelting showed that further increasing the temperature in fabricated specimens can lead to a higher relative density due to disassociation of oxides. However, preheating caused strong oxidation and sintering of the unfused powder, which restricts its reusability. Increased oxidation as a result of preheating was not indicated in the fabricated specimens. Thus, the positive influence of preheating, which can lead to the production of copper components in unique shapes with relative density greater than 99%, can outweigh the negatives connected to unfused powder waste.

6 CONCLUSIONS

This dissertation thesis deals with the application of preheating in LPBF technology. The thesis is divided into two research areas which deal with the application of preheating to decrease the residual stresses and processing of hard-to-process materials. Those two topics were identified, based on the review of the literature, as issues that hinder further technology utilization. The residual stresses limit the processing ability of some materials by LPBF and are the main cause of distortions, warping, and cracks. Thus, parts must be strongly fixed on the build platforms by support structures. However, the support structures prolong the build time, material waste, and post-process requirements. Thus, reduction of residual stresses, and consequently support structures, can lead to lower production costs. For that reason, it is important to study the techniques for their reduction. The main objective of the first topic was to experimentally analyse the influence of preheating on the Ti6Al4V and IN939 alloys.

The second topic identified as a perspective for the usage of preheating in LPBF was the processing of reflective materials. The preheating has an ability to decrease energy delivered from laser thus, it can be perspective for copper fabrication and materials with similar behaviour. Copper has excellent thermal and electrical properties, which in combination with design possibilities allowed by LPBF can lead to fabrication of unique components. However, the fabrication of fully homogeneous copper by LPBF is a challenge, and so far, fully homogeneous specimens from pure copper have not been produced. Thus, the main goal was to experimentally identify the appropriate combination of process parameters for the production of copper specimens with high relative density and to investigate the effect of high-temperature preheating.

The thesis contains original results that expand the knowledge of high-temperature preheating in LPBF. The results were confronted with studies of other research groups, and the possible usage and its limitations were stated. The main contribution of the thesis can be summarized into the following points:

- The application of preheating at 550 °C on the Ti6Al4V alloy can significantly reduce internal residual stress and increase relative density. However, even in combination with other process parameter, residual stresses were not fully eliminated.
- The application of preheating at 550 °C led to fast degradation of the unfused Ti6Al4V powder. The concentration of oxygen and hydrogen exceeds the ASTM B348 requirement limit for grade 5 titanium after one production run.
- In contrast, preheating of 400 °C led to increased deformations of IN939 specimens, thus, higher residual stresses.

- The hardness, tensile strength, and 0.2% proof stress of IN939 increased and the elongation at break decreased due to the application of preheating.
- High preheating temperature had a significant effect on the size and occurrence of the carbide phase of IN939. The occurrence and size of the carbide phase correspond to the results of increased residual stresses and mechanical properties.
- The build time had a significant effect on the evolution of the carbide phase of IN939. Longer build time resulted in larger size and the occurrence of the carbide phase. This effect was more significant in combination with higher preheating temperature.
- Rapid oxidation of the unfused IN939 powder due to the preheating temperature of 400 °C was not detected using the EDX and XRD methods.
- Copper can be fabricated with a relative density greater than 99% with an infrared fiber laser with a maximum laser power of 400 W and preheating of 400 °C .
- The relative density of copper over 99% was achieved just with application of preheating at 400 °C. Preheating at 200 °C led to maximum relative density of 96%.
- However, preheating to 400 °C caused rapid oxidation of the unfused copper powder. Thus, restricted its reusability. The increased oxidation of the fused material was not detected by EDX or XRD methods.

Regarding the scientific questions, the obtained knowledge can be summarized in the following remarks:

- Q1. No combination of process parameters and preheating up to 550 °C that would lead to the elimination of residual stresses of the Ti6Al4V alloy was found. Residual stresses decreased significantly when preheating at 550 °C was applied and the results indicated that even a higher energy density delivered by the laser would lead to further reduction of residual stress. However, preheating to 550 °C led to rapid degradation of the unfused powder. Therefore, this technique is not cost effective for the elimination of residual stresses. Thus, the hypothesis was **falsified**.
- Q2. Preheating of 400 °C, as a single changed process parameter, led to an increase in residual stresses of IN939 specimens. The increase of residual stresses was attributed to the evolution of the carbide phase. Therefore, the hypothesis that preheating can decrease residual stresses of IN939 was **falsified**.

- Q3. The preheating at 400 °C caused an increase of the relative density of copper processed by LPBF. The fabricated component reached a relative density greater than 99% while preheating was used at 400 °C. Compared to 200 °C preheating, the maximum measured relative density was 96%. Therefore, the hypothesis that the application of high-temperature preheating can lead to fabrication of copper specimens with a high relative density was **confirmed**.

7 LITERATURE

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AUTHOR'S PUBLICATIONS

Publications related to the topic of this thesis

MALÝ, Martin, Christian HÖLLER, Mateusz SKALON, Benjamin MEIER, Daniel KOUTNÝ, Rudolf PICHLER, Christof SOMMITSCH a David PALOUŠEK. Effect of Process Parameters and High-Temperature Preheating on Residual Stress and Relative Density of Ti6Al4V Processed by Selective Laser Melting. *Materials* [online]. 2019, **12**(6), 930. ISSN 1996-1944. Available at: doi:10.3390/ma12060930

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Other publications

HERNÁNDEZ-TAPIA, Laura, Azalia Mariel CARRANZA-TREJO, Adelia KASHIMBETOVA, Serhii TKACHENKO, Zuzana KOLEDOVÁ, Daniel KOUTNÝ, Martin MALÝ, Ladislav ČELKO a Edgar B. MONTUFAR. Microstructure of Selective Laser Melted Titanium Lattices and In Vitro Cell Behaviour. In: METAL 2021 - 30th Anniversary International Conference on Metallurgy and Materials, Conference Proceedings [online]. B.m.: TANGER Ltd., 2021, s. 1179–1185. ISBN 9788087294994. Available at: doi:10.37904/metal.2021.4259

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2020 – 2022, TAČR Zéta TJ04000314, Additive Manufacturing of Turbine Engine Components from Heat-Resistant Alloy Inconel 939, project leader

2020, FV 20-21, Innovation of the subject Additive Technologies in the application of design rules for components for 3D printing, project leader

2017 – 2020, ESA Contract no. 4000123317/18/NL/GLC/hh, Additive Design for Aerospace Applications Capabilities (ADAAC), team member

2019, FV 19-19, Innovation of the subject Additive Technologies in methods for the design of experiment, project leader

2017 – 2019, MPO FV20232, Structural biodegradable implants processing by means of direct metal laser sintering, team member

Teaching activities

- Additive Technologies (ZAT)
- Team Project (ZKP)
- Master Thesis Project – Results and Discussion (ZD5)
- Engineering Drawing Fundamentals (1K)
- Engineering Drawing (2K)
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Master and bachelor theses supervision

Bachelor theses

- 2020: Design of device for powder recoating at elevated temperatures for thin-walled parts
- 2021: Development of a strategy for 3D printing of copper using Laser Powder Bed Fusion
- 2021: Methods for surface improvement of components processed using Selective Laser Melting

Master thesis

2021 – 2022: Processing of Inconel 939 alloy using laser powder bed fusion at elevated temperatures

