Inconel 939 processed by selective laser melting: Effect of microstructure and temperature on the mechanical properties under static and cyclic loading

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Nickel-based superalloys, such as Inconel 939, are a long-established construction material for high-temperature applications and profound knowledge of the mechanical properties for this alloy produced by conventional techniques exists. However, many applications demand for highly complex geometries, e.g. in order to optimize the cooling capability of thermally loaded parts. Thus, additive manufacturing (AM) techniques have recently attracted substantial interest as they provide for an increased freedom of design. However, the microstructural features after AM processing are different from those after conventional processing. Thus, further research is vital for understanding the microstructure-processing relationship and its impact on the resulting mechanical properties. The aim of the present study was to investigate Inconel 939 processed by selective laser melting (SLM) and to reveal the differences to the conventional cast alloy. Thorough examinations were conducted using electron backscatter diffraction, transmission electron microscopy, optical microscopy and mechanical testing. It is demonstrated that the microstructure of the SLM-material is highly influenced by the heat flux during layer-wise manufacturing and consequently anisotropic microstructural features prevail. An epitaxial grain growth accounts for strong bonding between the single layers resulting in good mechanical properties already in the as-built condition. A heat treatment following SLM leads to microstructural features different to those obtained after the same heat treatment of the cast alloy. Still, the mechanical performance of the latter is met underlying the potential of this technique for producing complex parts for high temperature applications.

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1. Introduction

Nickel-based superalloys are intensively used in the aerospace and energy sector due to their excellent oxidation resistance and their outstanding creep properties at high service temperatures. Conventional casting techniques are limited with regard to the components complexity thus limiting the operating range and the efficiency. Hence new manufacturing processes, expanding the design possibilities, such as selective laser melting (SLM) have come into the focus of research. SLM is a technique which utilizes a high power laser beam to produce components layer by layer directly from a metal powder without need of further tools. This method allows for economical production of small batches and single components [1–8]. So far, stainless steel, nickel alloys and TiAl6V4 have been widely used for manufacturing parts by SLM and other solid freeform fabrication processes [9,10]. For applications in the temperature range up to 650 °C the nickel-based superalloy Inconel 718 is one of the most commonly employed materials [11]. Some previous studies already investigated the monotonic mechanical properties of samples made from this alloy using additive manufacturing (AM) processes such as SLM [12], laser solid forming [13], direct laser deposition [14], laser net shape manufacturing [15] and laser rapid forming [16]. Comparing SLM-processed to wrought material several authors observed similar or even better tensile properties at room temperature in case of SLM. The reasons are seen in smaller grains present after solution annealing and ageing treatment of the SLM-processed samples. Blackwell [14] also stated higher ductility after hot isostatic pressing due to improved interlayer bonding. However, for applications at higher temperatures other nickel-based superalloys are required. A promising candidate is Inconel 939, which already is used as a cast alloy in combustion turbines at operating temperatures of up to 850 °C. The alloy shows excellent oxidation resistance and high creep strength at elevated temperatures.
The excellent performance is caused by coherent $\gamma$-particles present after solution annealing and single-step ageing of the cast material [17]. However, since SLM is a layer-wise manufacturing process with repeated heat dissipation, it was questionable if this heat treatment, originally recommended for the cast alloy, would lead to the same microstructural features. Therefore, the effect of SLM on the microstructure and the resulting mechanical properties of Inconel 939 was investigated in this study. It is shown that the microstructure of the SLM-material can be modified by a subsequent heat treatment and that mechanical properties similar to conventional cast material can be achieved. The microstructural features in the as-built condition as well as after heat treatment were characterized and correlated to the mechanical performance under monotonic and cyclic loading at different temperatures. The findings are compared to the cast condition to evaluate the applicability of the SLM technique for producing highly complex Inconel 939 parts.

2. Experimental procedure

In the present study a SLM 250 HL machine (SLM-Solutions GmbH) equipped with an yttrium fiber laser with a power of 400 W was employed for fabrication of specimens from the nickel-based superalloy Inconel 939, cf. Fig. 1. The processing data were created via SLM AutoFab software (Marcam Engineering GmbH) on the basis of conventional CAD-Data of dog bone shaped samples with nominal gauge length dimensions as given in Fig. 2(d). The scan velocity varied between 540 and 620 mm s$^{-1}$ and the hatch distances were set between 0.15 and 0.12 mm, depending on which part of the sample was manufactured.

Powder material with an average particle size of 30 $\mu$m was deposited on pre-heated (100 °C) stainless steel substrate plates. The thickness of each layer was also 30 $\mu$m. Adding a 4 mm supporting structure underneath the actual specimens aimed at compensating the strain mismatches due to different thermal expansion coefficients between the steel substrate and the Inconel 939 and also allowed for an easy separation of the specimens from the substrate plate after manufacturing. In order to avoid contamination, the manufacturing process was carried out under argon atmosphere. The chemical nominal composition of Inconel 939 according to [17] is 0.15C, 22.4Cr, 19Co, 2W, 1Nb, 1.4Ta, 3.7Ti, 1.9Al, 0.12Zr, 0.01B and balance Ni, in wt%.

In order to examine the influence of the manufacturing direction on the mechanical properties, the samples were oriented either parallel or perpendicular to the building platform and the samples are referred to as 0° and 90°, respectively, in the remainder of the text.

A conventional cast Inconel 939 alloy served as reference material. Several samples of both the SLM-material as well as the cast alloy were heat treated in evacuated quartz glass tubes. According to parameters recommended for the vanes of land-based turbines, the heat treatment comprised a solution annealing for 4 h at 1160 °C followed by a single stage ageing for 16 h at 850 °C [17].

The specimens were either mechanically polished down to a grit size of 5 $\mu$m or tested in the as-built condition without any further surface treatment. Using an electron backscatter diffraction (EBSD) system, the grain orientation, grain size and grain boundary characteristics were determined. EBSD-scans were conducted in the gauge length of the samples (Fig. 2(d)). For this purpose mechanically pre-polished specimens were electropolished for 10 s under 20 V in a 5% perchloric acid solution. In order to examine dislocation characteristics, precipitates and local chemical compositions following the heat treatment, a transmission electron microscope (TEM) equipped with an EDS (energy dispersive X-ray spectroscopy) system operating at a nominal voltage of 200 kV was used. The TEM samples were taken from the grip section of the specimens (Fig. 2(d)) and mechanically polished down to 0.08 mm thin discs, which were then thinned by twin jet polishing at −22 °C under a potential of 21 V with a 5% perchloric acid solution.

Both tensile tests and low-cycle fatigue tests were carried out on a servo-hydraulic testing machine operating in displacement and strain control, respectively. A high-temperature extensometer was applied for measuring the strain. Heating was realized with a high-frequency induction heater. In order to avoid crack initiation at thermocouples welded on the sample surface, the temperature was controlled using a non-contact pyrometer. Solely for calibration of the pyrometer a thermocouple was spot welded onto the gauge section of a reference sample. All tests were conducted in ambient air. The test conditions for the fatigue experiments are summarized in Table 1.

3. Results and discussions

3.1. Microstructure

Fig. 1 shows optical micrographs of a SLM-processed and electropolished 90° specimen in the as-built condition. The images were taken parallel (a) and perpendicular (b) to the building direction (BD) as indicated in the images. Clearly visible are arch-shaped lines resulting from an equally shaped melt pool during processing (Fig. 1a). In contrast, in the plane perpendicular to the building direction these features appear elongated, which is related to the concurrent laser movement [12,13,18].

These features, however, have a minor impact on the microstructure with respect to grain orientation and morphology as shown in Fig. 2 by means of EBSD. Both the 0° as well as the 90°
sample feature columnar grains elongated alongside the building direction across several layers. This epitaxial grain growth during crystallization of SLM-materials leading to a strong bonding between the layers was also observed by several other authors [13,19] and is responsible for the invisibility of the arch-shape in these images. The grain aspect ratio (GAR) is lower in case of the 0 ° sample, i.e. the grains are thicker and shorter, leading to slightly different calculated average grain sizes of about 30 μm in case of the 90 ° sample and of 35 μm in case of the 0 ° sample. This is attributed to the heat flux during processing, which depends on the material amount surrounding the melt pool. It is already known that most of the heat is transported through the dense material, e.g. the solidified volume rather than the powder material [19]. In case of the 90 ° samples most part of the energy passes through the gauge section and is strongly directed towards the building platform, whereas for the 0 ° sample heat flow additionally is directed towards the grip sections as the sample is manufactured horizontally and consequently there is more material on two sides of the melt pool.

During ageing grain coarsening occurs and the grains lose their strictly columnar morphology as can be deduced by comparing Figs. 2 and 3. Several authors reported that recrystallization takes place in SLM-material during heat treatment, provided that the temperature as well as distortion energy are high enough [13,16]. Since the material is not subjected to any deformation during processing, the critical mechanical energy necessary for the onset of recrystallization is assumed to result from residual stresses typically caused by the high thermal gradients during SLM.

Comparing the different building directions, the 90 ° specimen features much finer grains than the 0 ° specimen (Fig. 3(a) and (b)). The average grain sizes are about 35 μm in case of the former and 70 μm in case of the latter. Since the heat treatment temperature was identical, this difference is attributed to higher residual thermal stresses present in the as-built condition of the 90 ° direction. This assumption could be supported by TEM as some images revealed higher dislocation densities for the 90 ° specimens. Still, the overall grain size remains small, which becomes obvious by comparing both SLM-processed conditions to the conventionally cast sample (Fig. 3(c)). Considering the different scales, a difference by a factor of at least 3 can be inferred.

Texture analyses were conducted by X-ray diffraction (XRD) measurements. As shown in Fig. 4 for a 90 ° specimen, a preferred orientation along the [001] direction with respect to the building direction is present in the as-built condition. Strongly textured microstructures were already observed in other studies and are related to grain growth dominated by heat flux [20,21,22]. In contrast, the texture is much weaker after ageing, which is attributed to the effect of recrystallization during heat treatment.

Microstructural insights on a much smaller scale were obtained by TEM (Fig. 5). Again elongated structures with low-angle boundaries oriented parallel to the building direction are present in the as-built condition of both directions, but these features have a much smaller scale and are much more periodical than the grains detected by EBSD. However, this pattern is not present throughout the entire specimen, as cell-shaped structures were also detected, as shown in Fig. 5(c), which was recorded from a different area of the TEM sample. Similarly, these structures were seen in SEM images using back scattered electron contrast (Fig. 5 (d)). This is opposing to observations made by Amato et al., who stated that the substructures are entirely directed towards the building direction and supported this by composited TEM images [12]. Thus, the formation mechanism as well as the distribution and orientation of these sub-structures are still not clear and will be subject to future work.

After ageing, TEM analyses reveal the presence of the hardening γ’-phase in both the SLM-processed as well as in the cast material (Fig. 6(a) and (b)). The corresponding selected area electron diffraction (SAED) patterns are shown in Fig. 6(e) and (f). Both patterns feature weak superlattice reflections resulting from the ordered crystal structure and no extra spots are
discernible. This confirms the coherent nature of the \(\gamma'\)-precipitates \([23]\). The chemical composition as determined by EDS is dominated by nickel with additional amounts of titanium and aluminum, in line with Hoffelner and Tschegg who designated these as (Ni,\ldots)\(_3\)(Al,Ti,\ldots) \([24]\). Although the particle morphology and distribution in the matrix are similar, the size is slightly higher in case of the SLM-processed material. The SAED pattern corresponding to Fig. 6(d) cannot be discerned from the one shown in Fig. 6(e) and thus, is not duplicated for the sake of brevity. In addition, brittle phases are present in the aged SLM-processed condition, as visible in Fig. 6(c). These are typically referred to as topologically closed packed (TCP) phase or as hexagonal \(\eta\)-phase \([25–28]\). Whereas the morphology of the former is needle like featuring a chromium rich composition, the latter is slightly wider and has a nickel-based composition. The morphology can be discerned in Fig. 6(c) and the differences in chemical composition were confirmed via EDS-measurements conducted for both, the smaller vertical as well as the broader horizontal features. Fig. 6(g) shows the corresponding SAED pattern with additional reflections resulting from these two incoherent phases. The orientation difference visible in Fig. 6(c) can be found again as indicated by the blue and green lines in Fig. 6(g). Usually, these undesired brittle precipitates develop during long time operation in cast and wrought material \([29]\). However, the SLM-processed condition already features \(\gamma'\)-precipitates after solution annealing (Fig. 6(d)). Similarly, Amato et al. observed such precipitates in recrystallized areas of SLM-processed Inconel 718 after annealing \([12]\). This is in strong contrast to equally treated cast material, which is supposed to be fully solution annealed after 4 h at 1160 °C. These observations indicate that the repeated heat input during the SLM process is sufficient for forming nuclei of precipitates, thus facilitating and accelerating their full development during the subsequent heat treatments. Interestingly, the substructures, which were visible directly after the SLM process, disappeared in course of the two stage heat treatment (cf. Figs. 5(a) and (c) and 6(c)).

### 3.2. Mechanical characterization

The deformation behavior under tensile load is depicted in Fig. 7. Tensile testing was conducted for 0° and 90° samples at room temperature (RT) in the as-built and in the aged condition. Additional tests were run for the 90° samples after solution annealing and at a temperature of 750 °C.

Comparing the 0° and 90° specimens tested at RT in the as-built condition, the latter clearly provides the better performance in terms of ductility, while the yield strength is almost equal. After ageing this relation changes, and the 90° specimen is then characterized by higher yield strength and the ductility is diminished. Relating these observations to the microstructural conditions, it becomes clear that several aspects influence the mechanical performance. The elongated grains revealed in the EBSD micrographs (Fig. 2) and the preferred (001) orientation with respect to the BD (Figs. 2 and 4) should have a strong influence on
the mechanical properties of the samples tested in 0° and 90°, leading to substantial differences in strength and ductility. Indeed, elongation at failure is varying almost by a factor of two in the as-built condition (Fig. 7). This may be attributed to the different mean free paths of dislocations induced by the difference in grain aspect ratio and the orientation of the grain long axes with respect to the loading direction. Interestingly, yield strength and ultimate strength of both sample orientations are very similar indicating that another microstructural feature has a strong impact on the strength values of the material. As shown in Fig. 5, all samples are characterized by the presence of substructures, e.g. low-angle grain boundaries, eventually acting as barriers for dislocation motion. Both sample orientations revealed two different kinds of substructures, i.e. elongated and equiaxed cells, both of sub-micron size. These substructures seem to mainly influence the strength of the SLM-processed IN939, such that the similarity of the structures in all samples leads to identical behavior in the early stages of deformation. In part, these findings are opposing the results of Vilaro et al. who reported a low ductility for 90° nickel-based and Ti6Al4V SLM-samples [19,30]. They attribute this to defects between layers, which get opened by the load. However, in the present study there were no such defects observed. Instead a stable bonding between the layers was realized by the processing parameters used.

In course of the two stage heat treatment, the residual stresses were annealed-out, the substructures got dissolved and recrystallization set in, leading to a finer grain morphology of the 90° specimen compared to the 0° specimen. Thus, the dominating effect of the substructures disappeared and yield strength became more dominated by the grain size as dictated by the Hall–Petch relation [31]. Consequently, the observed yield strength was higher in case of the 90° specimen. Concurrently, the precipitates, which evolved in course of ageing, led to an overall increase of the yield and ultimate strength for both directions and to a reduction of ductility as compared to the as-built condition. This effect is due to a reduced dislocation mobility based on the finely dispersed precipitates, as discussed in detail in [19].

The solution annealed 90° sample is characterized by yield strength and ductility values lying in between the as-built and the aged condition. The TEM image shown in Fig. 6(d) suggests that γ’-precipitates initially evolved during solution annealing but were not yet fully formed.

At higher testing temperatures the as-built as well as the solution annealed 90° samples show a clearly decreased ductility. The elevated temperatures during testing probably led to significantly accelerated forming of precipitates and thus embrittlement of the specimens.

For comparison, the mechanical performance of the cast material is given in the inset in Fig. 7. With yield strengths from about 500 MPa to 800 MPa and ultimate strengths from 750 MPa to 950 MPa the cast material features inferior strength values compared to the SLM-processed material. Likewise the ductility is significantly lower in the initial state. These differences are attributed to the smaller grain sizes present in the SLM-processed material (Fig. 3). Similar effects were also observed for numerous other alloys, e.g. for an ultrafine-grained NbZr alloy, where the smaller grain sizes led to both an increase in yield strength and a higher ductility due to a more homogeneous deformation [32]. These assumptions are supported by hardness measurements conducted on both materials yielding values of 391 HV0.5 for the cast and of 450 HV0.5 for the SLM-processed material. For the cast material the elongation to failure is low in
the aged conditions at both room temperature and at 750 °C. The ductility of the as-cast material is higher at elevated testing temperature than at room temperature, which is contrary to the observations made for the SLM-processed material. This is, however, not surprising, when taking the microstructural conditions into account. Nuclei for precipitations already evolved during SLM and lead to fast formation of precipitations during monotonic testing at high temperatures, which results in high brittleness. As these nuclei are not present after casting, the formation of precipitates is initially hindered. Therefore embrittlement does not occur and the ductility is increased in comparison to the same condition tested at RT.

Jahangiri et al. investigated aged wrought Inconel 939 and compared the microstructural and mechanical behavior to equally aged cast material [33]. Although the heat treatment parameters were different to the present study, similar observations can be made. Thus, higher strength occurred from RT to 850 °C in the wrought material, due to smaller grains sizes and, in addition, to the presence of twins. However, comparing the strength from the aged wrought and the aged SLM-processed material the strength

Fig. 6. TEM bright-field images showing (a) fine γ’ precipitates in the aged SLM-processed material; (b) fine γ’ precipitates in the aged cast material; (c) brittle phases in aged SLM-processed material; (d) precipitates after solution annealing of SLM-processed material; (e), (f) and (g) depict the SAED pattern obtained from small areas in (a), (b) and (c), respectively. The blue and green lines in (g) highlight the reflections from two phases oriented at an angle α of about 70° to each other, which can be found again in (c). (For interpretation of references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 7. Monotonic stress strain curves for Inconel 939 in different microstructural conditions after SLM and casting.
of the latter is still not met as the grain size of the wrought material remains relatively high. Regarding the ductility, again the SLM-processed material has an inferior elongation due to the presence of the brittle phases, which already evolve during ageing. However, these results clearly reveal the potential of SLM-processed material for applications where excellent yield strengths up to high temperatures are required.

The fatigue lives of the here tested conditions are summarized in Table 2. Comparing the initial states (as-built, as-cast), a significantly higher fatigue life at RT in case of the SLM-processed specimen can be deduced, although the surface was not polished and thus featured a higher roughness. This difference is explainable by the corresponding yield strengths, which were observed in the monotonic tests. The cyclic strain amplitude of 0.5% leads to stress amplitudes of about 700 MPa. Thus, the yield strength of the as-cast sample is clearly exceeded resulting in significantly higher plastic strain amplitudes, and consequently earlier failure as compared to the SLM-processed sample. This aspect is obvious from the hysteresis loops shown in Fig. 8. The findings are supported by additional tests conducted with a lower total strain amplitude of 0.35%, where similar fatigue lives in both conditions resulted (data not shown).

After ageing different trends were observed. The fatigue life of the SLM-processed sample was reduced by ageing, whereas the cast sample revealed a higher fatigue life. In case of the former, the precipitates and the concomitant brittleness lead to higher sensitivity to crack initiation and crack growth, thus to lower fatigue life, even when the surface was polished. In case of the latter the precipitates result in a higher yield strength, as shown in Fig. 7. Consequently, the resulting plastic strain amplitude during cyclic testing is reduced and a higher fatigue life than in the as-cast condition is achieved. The lower fatigue life of the aged SLM-processed sample in comparison to the aged cast sample is explainable by process-induced defects, such as pores typically present in additively manufactured material. These act as local stress raisers and consequently reduce the loading capability of the brittle microstructural condition, especially under cyclic loading conditions [34].

At elevated temperatures, all conditions feature reduced fatigue lives. The most striking difference, amounting to almost 4500 cycles, occurs in case of the SLM-processed as-built condition. This again is related to the fast formation of precipitations during testing, as discussed above. However, the overall pronounced reduction of the fatigue lives is rather based on an enhanced dislocation mobility, as typically present at high temperatures. Since only few tests were conducted no assertions can yet be made with respect to scatter resulting from variability of the SLM process. Thus, further studies are needed in order to fully evaluate the potential of SLM-processed Inconel 939 under cyclic loading conditions. However, when an appropriate heat treatment is employed, the results presented in this study already reveal that the performance of SLM-processed IN939 is similar to cast material under the current test parameters.

4. Conclusions

In this study the microstructure of SLM-processed Inconel 939 was investigated and related to the mechanical performance at RT and at 750 °C under monotonic and cyclic loading. The microstructural conditions encompassed as-built, solution annealed and aged material. The microstructural and mechanical differences to conventionally cast material were discussed.

The main findings can be summarized as follows:

- Processing of Inconel 939 by Selective Laser Melting results in formation of a textured microstructure with fine columnar grains across several layers and micro-scaled substructures.
- The conventional two-stage heat treatment leads to recrystallization of the SLM-processed material. Still the grain sizes remain significantly lower than in the cast alloy.
- The mechanical performance under monotonic load of the as-built condition is characterized by high ductility irrespective of

Table 2

<table>
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<tr>
<th>Condition</th>
<th>SLM-processed, as-built</th>
<th>SLM-processed, aged</th>
<th>Cast, as-cast</th>
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<td>1598</td>
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<td>230</td>
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Fig. 8. Hysteresis loops for the cast IN939 and SLM-processed as-built IN939. Both tests were conducted at RT at a constant strain rate of 0.006 s$^{-1}$ and a total strain amplitude of 0.5%. Hysteresis are plotted at half life.
the building direction. By ageing, the yield strength is increased and embrittlement sets in due to formation of precipitates.

- In all conditions the yield strength of the SLM-processed material is higher than that of the cast material. This is caused by smaller grains and the substructures present in the microstructure after SLM.
- In its initial SLM-processed condition IN939 shows significantly better performance under cyclic loading at room temperature than the cast material. This is attributed to lower energy dissipation per cycle.
- Testing at high temperatures leads to fast formation of precipitates and thus embrittlement of the SLM-processed as-built condition. This suggests that nuclei already evolve due to high temperatures and repeated heat dissipation during the SLM process. Both the ductility as well as the fatigue lives are considerably reduced.

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References

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