Temperature-Dependent Dynamic Strain Aging in Selective Laser Melted 316L

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Additively manufactured austenitic stainless steel AISI 316L (EN 1.4404, X2CrNiMo17-12-2) is used at higher temperatures, e.g., in space applications. However, the high-temperature properties of such materials have not been analyzed in detail yet. Thus, selective laser melted (SLM) 316L is tested in the solution-annealed condition by compression and tensile tests at temperatures between 25 and 877 °C. The compressive strength of SLM 316L is higher in comparison with the conventionally produced reference material due to hardening by a high dislocation density and a fine substructure. However, tensile tests reveal a loss in ductility of the SLM material at temperatures between 300 and 627 °C, where the elongation to fracture is reduced from 65% to 39%. Alloying elements cause serrated yielding in the affected temperature range. Together with an increased normalized work-hardening rate and a negative strain rate sensitivity, dynamic strain aging is found to cause the reduction of ductility.

1. Introduction

Additive manufacturing (AM) has received increasing attention for the production of complex parts for industrial applications during the past years. Although many machine manufacturers promise excellent mechanical properties of their selective laser melted (SLM) 316L alloy at room temperature, proven by numerous publications,[1–5] far lower attention has been devoted to the high-temperature properties of 316L. In contrast to conventionally produced 316L, AM 316L is additionally strengthened by fine dislocation cells, which dissolve between 600 and 1000 °C.[6] Due to that, superior tensile properties of AM 316L were also obtained at temperatures of 250[7] and 800 °C.[8] For long time exposures under high temperatures, however, unstable dislocation cells can deteriorate the creep properties.[9,10] Saeidi et al.[8] also observed the propagation of Luders bands, which are closely related to dynamic strain aging (DSA), at 800 °C. However, no systematic study on the influence of the SLM microstructure on the mechanical properties over the entire temperature regime from room temperature to 900 °C has been conducted yet. This is surprising, as AISI 316L is a widely used austenitic stainless steel for applications at elevated temperatures, and it is known from the conventionally produced material that DSA occurs at elevated temperatures.

The improvement of the process parameters in recent years with respect to the laser power, hatch distance, scan strategy, etc. allows nowadays the manufacturing of geometrically accurate parts with high strength.[11] The high thermal gradients and high solidification rates in the SLM process lead to a fine dendritic solidification with microsegregation. Rapid local heating and cooling introduce significant stresses in accordance with the temperature gradient mechanism (TGM).[12] Recent results of Bertsch et al.[13] showed that these stresses and strains contribute strongly to the formation of dislocations. Mainly due to the high dislocation density, networks are formed. These dislocation cells typically overlap the microsegregations. Dendritic segregations and dislocation cells are known to be beneficial for the mechanical properties.[1–3,5]

The main strengthening mechanisms of the austenitic steel 316L are solid solution and Hall–Petch strengthening. In the case of SLM-processed 316L, additional strengthening by the dendritic substructure has to be considered. These substructures consist of segregated elements and entangled dislocations contributing to the strength of the material.[1,4–15] One model to understand the fundamentals of strengthening by cellular dislocation structures was established by Mughrabi.[16–18] The so-called composite model describes the stress distribution in the soft cell and hard wall regions and enables the calculation of the total strengthening effect by a modified Taylor equation, using a geometric constant for the heterogeneous composite, \( \alpha_{het} \), depending on the wall thickness and cell diameter.[18] It was also found by Blum and Reppich[19,20] for cellular dislocation networks developed under static creep loads, that the...
strengthening contribution is inversely proportional to the cell diameter \( (\sigma = kGb/d_{\text{cell}}) \). Gallmeyer et al.\[^{15}\] found that the calculation of the dislocation density on the basis of the cell diameter of 620 nm is nearly equal to the dislocation density obtained by counting dislocations in transmission electron microscopy (TEM) images. By the application of Taylor’s equation, both result in a strengthening contribution of 297 MPa for as-built IN718. Additively manufactured 316L exhibits in the as-built condition a yield strength (YS) between 427 and 640 MPa due to the described various strengthening mechanisms.\[^{21–23}\] Subsequent solution heat treatments lead to dislocation annihilation and the homogenization of the dendritic segregations. As a result, a lower YS between 364 and 436 MPa\[^{8,22}\] is obtained. However, heat treatments are often conducted to reduce residual stresses and distortions after the removal of the building platform.\[^{24}\]

Although the 316L alloy exhibits a high corrosion resistance and good mechanical properties at high temperatures, it is known that the conventionally produced 316L is affected by DSA, compare other studies\[^{25–51}\]. At intermediate temperatures, the diffusion velocity of solute atoms is similar to the velocity of dislocations, resulting in the hindering of dislocation movement. This causes locking and avalanche-like unlocking of dislocations, leading to serrations in the stress–strain curve at temperatures between 250 and 600 °C.\[^{52}\] This effect is called after Portevin and Le Chatelier (PLC effect), who first described this phenomenon.\[^{32}\] In addition, the mechanical properties are influenced. Besides a slight increase in the strength, 316L shows a huge ductility decrease in this temperature range.\[^{25,27,33}\] The magnitude, however, varies with the applied processing technologies of 316L. Thus, it is important to investigate the properties of SLM-processed and solution-annealed 316L from room temperature to elevated temperatures to analyze possible differences between additively manufactured and conventionally produced 316L and evaluate its potential for future applications in engine propulsion systems in Ariane rockets. Therefore, the microstructure and its influence on the compression and tensile strength is analyzed and discussed here.

2. Experimental Section

2.1. Manufacturing of Samples

AM of 316L samples was conducted on an M 290 (EOS GmbH, Krailling, Germany) SLM machine at Ariane Group GmbH (Taufkirchen, Germany). The composition of the 316L powder is shown in Table 1. The build-up process was conducted with a layer thickness of 40 μm and the usage of the standard 316L process parameters of EOS including a rotating stripe exposure strategy. Herein, the calculated laser energy density was, in accordance with the study by Song et al.\[^{14}\] 2.7 J mm⁻². The 316L powder had a particle size between 20 and 65 μm and was supplied by OC Oerlikon Corporation AG (Pfäffikon, Switzerland). Samples for microstructural investigations were cut out of a part built up in the center of the building platform. The coordinate system was defined as the following: x-direction points to the right side and y-direction to the back side of the SLM machine and building platform. The z-direction was defined as building direction. As a reference material, a conventionally produced rod of the same steel with a diameter of 10 cm was investigated.

2.2. Heat Treatment

All additively manufactured samples were subjected to heat treatment in a vacuum furnace prior to microstructural and mechanical characterization. The material was heated to 1050 °C and held for a duration of 1 h. The conventional material was delivered in a heat-treated condition.

2.3. Sample Preparation and Characterization

Samples for microstructural analysis were ground down to a grit size of 2500, mechanically, and electrochemically polished using a 3 μm diamond suspension and an electrolyte A2 (Struers GmbH, Willich, Germany). The microstructure was analyzed with a scanning electron microscope (SEM) Zeiss Crossbeam 540 (Oberkochen, Germany) using backscattered electron (BSE) contrast and an electron backscattered diffraction (EBSD) detector (Oxford Nordlys nano). The EBSD measurements with 1000 × 750 pixels were carried out at 20 kV, with a binning of 4 × 4 and a step size of 1 μm. For the further processing and evaluation, the software Channel 5 (HKL) was used. Fine lines in inverse pole figure (IPF) mappings indicated low-angle boundaries with misorientation between 2° and 15° and thicker lines high-angle grain boundaries with angles above 15°. The elemental composition of interdendritic regions was determined by energy-dispersive X-ray spectroscopy (EDS) (Oxford X-Mat 150).

2.4. Mechanical Testing

Tensile and compression tests were conducted at temperatures of 25, 300, 627, 777, and 877 °C. Additional compression tests were conducted at a temperature of 350, 400, 500, and 677 °C. The loading direction was parallel to the building direction. The compression tests were conducted on an Instron 4505 (Instron GmbH, Darmstadt, Germany) testing machine, equipped with a Hegewald and Peschke (Meß- und Prüftechnik GmbH, Nossen, Germany) control unit. Therefore, vertically built SLM rods with a diameter of 12 mm were turned to 4 mm and divided into several samples, each with a length of 7 mm (see Figure 1a). The conventional reference material was machined by spark erosion. Both types of cylindrical samples were ground down to a length of 6 mm up to a grit size of 2500 to parallel the flat surfaces. The high-temperature tests were conducted in a three-zone furnace. The tests were controlled by the true strain rate, obtained from a linear variable differential transformer (LVDT). The initial strain rate was 10⁻³ s⁻¹. Several strain rate jumps to 10⁻⁴ and 10⁻⁵ s⁻¹ were conducted.
after achieving certain levels of strain. The strain rate sensitivity (SRS) \( m = \frac{\partial \ln(\sigma)}{\partial \ln(\dot{\varepsilon}/\dot{\varepsilon}_0)} \) was determined at the first and second transition in accordance to the schematic drawing in Figure 1a. Therefore, the stress was fit with a straight line and interpolated to the related strain where the jump in strain rate was applied. In addition, the stress difference within the plastic strain interval from 5% to 0.5% strain and the normalized work-hardening rate was calculated by the equation \( \theta = \frac{\sigma_{\text{pl},0.05} - \sigma_{\text{pl},0.005}}{0.045}/E \). The temperature-dependent Young’s modulus provided by the tensile tests was used for the calculations. The values were related to the true stress \( \sigma \) and true plastic strain \( \varepsilon_{\text{pl}} \).

The tensile tests were conducted on a Zwick 1456 (ZwickRoell GmbH & Co. KG, Ulm, Germany) testing machine in accordance with DIN 6892. The samples had a gauge length of 35 mm and a diameter of 6 mm (see Figure 1b). Each sample was heated and held at the desired testing temperature for 1 h before starting. Three samples were tested per temperature. The engineering strain rate was set to \( 10^{-3} \text{s}^{-1} \). The normalized work-hardening rate \( \theta \) was calculated analogously to the compression tests. The Young’s moduli were calculated by the slope of the first loading of the sample.

### 3. Results

#### 3.1. Microstructure of Additively and Conventionally Manufactured Material

The microstructure of the conventional reference material is shown in Figure 2. The EBSD mapping reveals randomly oriented grains with an average grain size of 30.1 ± 17.0 μm. Most of the grains have a polyhedral shape and many twins are present. Grain boundaries are frequently decorated with phases that appear bright in the BSE contrast and have lengths of up to several ten micrometer. Additional EDS analysis revealed an enrichment of Cr and Mo in that phase. In accordance with the literature, the phase was determined as δ-ferrite due to its composition, shape, size, and formation at grain boundaries. The phase precipitated during solidification and although it should be possible to eliminate the remaining δ-ferrite by long-term heat treatments, this is often not conducted due to economic reasons.

Figure 3a shows a faded columnar and cellular dendritic substructure in the additively manufactured 316L resulting from the high thermal gradients, solidification velocity, and subsequent solution heat treatment. The heat-treatment procedure dissolved the interdendritic segregations of heavier elements or ferrite-stabilizing elements (Mo, Cr). The diameter of the remaining dendritic cells lies between 0.5 and 1 μm. In an as-built condition, the dendritic substructure usually goes along with a high dislocation density at the cell boundaries resulting from high thermal stresses. It can be assumed that heat treatments between 800 and 1000 °C lead to a reduction in the dislocation density and the residual stresses. A higher-resolution image (Figure 3b) of the heat-treated condition shows an area where the substructure has vanished and left parallel line patterns (see arrow). In accordance with the studies by Man et al. and Kong et al., the still visible patterns at the cell boundaries are...
due to dislocations. TEM investigations show the rearrangement of formerly entangled dislocations to ordered patterns at the remaining cell boundaries after the heat treatment.\textsuperscript{[39,41]}

Aside from the almost vanished substructure, the heat-treated SLM material is fully unrecrystallized and exhibits still an anisotropic microstructure due to the layer-wise manufacturing process. Figure 3c shows the grain structure in the plane perpendicular to the building direction (xy plane). The crystal orientation is coded by the IPF coloring with respect to the sample normal. The rotating stripe scan strategy caused the growth of grains with the alignment of the $<101>$ crystal direction along the building direction (green color in Figure 3c). These $<101>$-oriented grains are often surrounded by smaller $<001>$-oriented grains. Due to the high area fraction of the coarser grains, a predominant texture of the $<101>$ direction along the building direction can be found. The scan strategy with its rotation after each layer leads also to a stripe pattern because of the alternating hatch vectors and its rotation by around $67^\circ$ after each layer. The coordinate planes parallel to the building direction, as shown exemplarily for the yz plane, exhibit V-shaped grains (Figure 3d). The grains are predominantly oriented with their $<001>$ crystal axis along the $x$-direction of the manufacturing process. As Keshavarzkermanii\textsuperscript{[42]} revealed, there may be a slight difference between both planes (xz and yz) that are parallel.

Figure 3. Microstructure and grain structure of heat-treated SLM 316L in a) partly vanished dendritic substructures and in b) residual substructure cells at high magnification. c–e) Results of EBSD analysis. Reference direction for IPF coloring of each map is normal to the displayed plane. c) The $xy$ plane exhibits a texture along the $<101>$ direction. The $yz$ plane exhibits a slight predominant $<001>$ texture along the $x$-direction. e) The contour plots describe the corresponding frequency distribution. f) The grain size distribution shows the differences between the two planes of the SLM material in comparison with the conventional reference material.
to the building direction of the heat-treated SLM material. In comparison with the earlier described conventional material, the heat-treated SLM material exhibits a high content of low angle grain boundaries, irregularly shaped grains, and no twins. Although the heat-treatment temperature of 1050 °C was held for 1 h, no significant recrystallization occurred. Figure 3f shows the high-angle grain boundary size distributions of the two different planes of the SLM material compared with that of the conventional material. The average grain sizes of the conventionally and additively manufactured material seem to be similar, but clear differences can be found in the size distribution. The distribution of the SLM material is much broader, due to the high amount of very small and very big grains. In addition, the irregular shape of the grains indicates that much more grain boundaries are present in the additively manufactured material, which might contribute to the strength by Hall–Petch hardening.

3.2. Compressive Strength and Serrations

Compression tests with strain rate jumps were conducted to analyze the strength, flow behavior, work-hardening rate, and SRS of the additively and conventionally produced 316L. Figure 4a,b shows the stress–strain curves of the heat-treated SLM material and conventional material from room temperature to 877 °C. The strength of the SLM 316L is superior to that of conventional 316L at all tested temperatures, which is shown in Figure 4c1. In the temperature region between 300 and 500 °C, the stress–strain curves of the conventional material are almost on the same level. Here, the strength loss with increasing temperature is less pronounced or even reversed (400 °C) in contrast to temperatures below 300 °C and above 500 °C, where an increase in temperature goes along with a huge loss in strength. In contrast to that, the heat-treated SLM material exhibits a steady decrease in strength between 300 and 500 °C. Also, the strength difference at a true plastic strain from 5% to 0.5% reveals a different behavior of the SLM and conventional material (Figure 4c2). The strength difference indicates the amount of work hardening. The interval from 5% to 0.5% was chosen to calculate the normalized work-hardening rate, which will be compared with that of tensile tests and literature data in Discussion. The strength difference decreases with increasing temperature for typical face-centered cubic (fcc) materials due to increasing recovery. However, the strength difference of SLM 316L remains on a constant level till 627 °C. In contrast, the conventional 316L exhibits

Figure 4. Results of compression tests with strain rate jumps conducted between 20 and 877 °C. Representative engineering stress (strain curves of a) heat-treated SLM and b) conventional 316L material. c1) The average strength at a true plastic strain of 0.5% is shown for the heat-treated SLM material and conventional 316L. c2) The strength difference in the strain interval between 5% and 0.5%. d) The occurring serrations at the low strain rates can be seen in the magnified view for both materials.
a significantly higher work-hardening rate between 327 and 677 °C. Both conditions show a high decrease in the work-hardening rate above 777 °C following the typical material behavior. At 877 °C, no differences in the strain-hardening behavior between the SLM and conventional material can be seen. Both material conditions show, especially in that temperature region, serrated stress–strain curves at low strain rates. Figure 4d shows a magnified view of separated stress–strain curves around the second strain rate jump from $10^{-4}$ and $10^{-5}$ s$^{-1}$. In accordance with the classification of serrations, the small oscillating serrations at a strain rate of $10^{-4}$ s$^{-1}$ are identified as type B serrations.$^{[31]}$ These serrations occur equally in both materials up to 627 °C and they are less pronounced at a strain rate of $10^{-5}$ s$^{-1}$. However, the conventional material is affected by serrations causing large yield drops at 300 °C. These type A (strengthening + yield drop) or type C (only yield drop) serrations appear again as type B and C serrations at 500 and 627 °C. In contrast to that, the curves of the SLM material exhibit almost no serrations in the lower-temperature region from 300 to 400 °C. However, even larger yield drops (type C) occur in the upper-temperature region from 500 to 627 °C. Above 677 °C, only small serrations were observed in both materials.

3.3. Tensile Properties: Ductility and Strain Hardening

Additional tensile tests were conducted on the heat-treated SLM material at a constant strain rate of $10^{-3}$ s$^{-1}$. The stress–strain curves are shown in Figure 5. The elongation to fracture decreases at temperatures between 300 and 627 °C, leading to a ductility minimum of the material. Tensile tests conducted at 627 °C show a jerky flow of the stress–strain curves. As shown in the magnification of the curve (Figure 5), the serrations can be attributed to oscillating type B serrations combined with stress drops of type A and C serrations in accordance to the study by Rodriguez et al.$^{[31]}$ At low strains, e.g., left inset, these serrations seem to strengthen the material as the serrations show an increase in the strength above the general level of the curve. Therefore, they can be attributed to type A serrations.$^{[31]}$ At higher strains, where no strain hardening occurs, the serrations (see right inset) are rather load drops, which decrease the stress without prior strengthening (type C).$^{[31]}$ The appearance of the PLC effect at high strains is also affected by the general decrease in stress due to an increasing damage in the sample. However, the PLC effect is still clearly distinguishable. At 627 °C, a high variation of the elongation to fracture was measured.

4. Discussion

4.1. Hardening Mechanisms of SLM 316L

The YS of the additively manufactured and subsequently heat-treated material is consistently higher than that of conventionally produced 316L independent of compressive or tensile loading, see Figure 6. In addition to solid-solution strengthening, the heat-treated SLM 316L is also strengthened by a high dislocation density and fine grains. Our data for the conventional material fit quite well to the data of Choudhary et al.$^{[25]}$ Apparently, the hardening effects by the high dislocation density and the substructures are still active in the heat-treated SLM material and this effect does not vanish at the test temperatures within the relatively fast test duration of about 2 h. However, it is likely that further recovery occurs during application at high temperatures and the YS of the heat-treated SLM material approaches that of the conventional material. This can already be seen for the highest test temperature of 877 °C, where a higher loss in strength was determined in the SLM material compared with the conventional material. As described in Introduction, the combination of high-temperature gradients and high solidification velocities generated a fine columnar dendritic microstructure in the SLM material, which contributes by dislocation strengthening in addition to solid solution and Hall–Petch strengthening to the strength of the material. It is clear that the strengthening of the dislocation cells is huge in the as-built condition, as TEM analysis in several publications showed the entanglement of dislocations with densities of about $1 \times 10^{14}$ m$^{-2}$$^{[15,38,43,44]}$. It is known that recovery...
and coarsening of the cells occur with increasing heat-treatment temperature. [46] However, these cells still contribute to the higher strength of the SLM material even after heat treatment at 1050 °C for 1 h, as the comparison with the strength of the conventional material showed. The relieving of residual stresses is essential for industrial applications to avoid distortion and the formation of cracks in parts after the manufacturing process. [45] Although the dislocation strengthening vanishes, EBSD measurements showed that no significant grain coarsening or recrystallization occurred during heat treatment. Thus, the strengthening effect of the fine grains remains also during the test procedure at high temperatures. The strengthening contribution might be higher compared with the conventional material due to the irregularly V-shaped grains that result in an increased grain boundary length per area. Due to that, the calculation of the strengthening contribution based on the equivalent grain diameter, used in the Hall–Petch equation (σ = k × d−0.5), might underestimate the real strengthening contribution of grains in the SLM material. In addition to the compression tests, tensile tests showed that the YS is superior and the ductility is equal to that of conventionally produced solution-annealed 316L at all temperatures. [25] The high ductility of AM 316L can be a result of deformation twinning as reported by Shamsujjoha et al. [46] The room temperature strength and ductility meet the requirements of annealed 316L (1.4404) according to DIN EN 10088-3. Thus, compression and tensile tests showed the effectiveness of dislocation and grain boundary strengthening in the SLM material in contrast to the conventionally produced material.

4.2. Effect of DSA

Regardless of the additional strengthening of the heat-treated SLM material by the earlier discussed remaining substructures, the SLM material is similarly affected by the DSA at certain temperatures as the conventional material. At temperatures above 200 °C, the diffusion velocity of solute interstitial atoms like C or N becomes similar to the velocity of moving dislocations. At higher temperatures, the diffusion rate of substitutional elements like Cr and Ni is also similar to the velocity of moving dislocations. [25,28,47–49] As a result, the dislocations can be pinned by both types of diffusing atoms, and a higher external stress must be applied to deform further. Besides the serrated yielding, DSA has a huge influence on several macroscopic mechanical properties. On the one side, the major drawback of DSA is the loss of ductility in the affected temperature range. On the other side, the repeated locking of dislocations causes a strengthening of the material. Both aspects will be discussed in the following sections.

4.2.1. Ductility Loss

For technical applications, the ductility of the material is of great importance. The observed huge loss in ductility at intermediate temperatures can be attributed to the embrittlement effect of DSA. [29,51] As shown in Figure 7a, the elongation to fracture over the temperature can be clearly divided into three different regions. The elongation to fracture decreases in the first region from 25 to 300 °C, from 65% to the plateau of about 40% between 300 and 627 °C. At temperatures above 650 °C, the ductility increases again due to the increased mobility of dislocations due to climbing and cross slip mechanisms. [25] Former investigations on conventionally produced 316L by Choudhary et al. [25] Tak et al. [26,27] and Brnic et al. [33] revealed elongations to fracture at these temperatures similar to those shown in Figure 7a. The different levels of elongations to fracture stem from different conditions of 316L that were tested.

This loss of ductility can be attributed to the effect of the SRS, as shown in Figure 7b, which was determined from the strain rate jumps in the compression tests. For fcc materials the SRS typically increases with increasing temperature. However, a decrease in the SRS is detected for the additively manufactured as well as for the conventional material from 25 to 500 °C. Negative SRS values were obtained in the temperature range from 300 to 627 °C with a minimum at 500 °C. SRS values, obtained at the strain rate jump from 10−3 to 10−4 s−1 (not shown here), show a shift to higher temperatures but the same

Figure 7. a) Influence of the DSA on elongation to fracture in the temperature range between 300 and 627 °C and its comparison with literature data on conventional 316L in different conditions: Choudhary et al. [25]: solution-annealed 316L(N), Tak et al. [26,27]: 316L cold worked with ε = 17%, and Brnic et al. [33]: unknown condition of 316L. b) Temperature dependency of the SRS of the heat-treated SLM and conventional material.
trend. The transition to negative values occurs at 400 °C and back to positive values at 677 °C. The origin of these negative SRS is a result of the increased pinning of dislocations at slower strain rates by the diffusion of the atoms. As known from literature, low or negative SRS is detrimental for ductility. May et al. observed an enhanced ductility of ultrafine grained material in comparison with conventionally grained material due to an increased SRS at higher temperatures. This is in good accordance with Woodford and Ghosh who showed experimentally and analytically that the necking of samples and thus the total elongation strongly depend on the SRS. If the SRS becomes negative, small strain localizations at heterogeneities are amplified due to the lower strength at the resulting higher strain rates, and thus fast necking of the specimen can occur at low strains. This means that if the SRS is low, only the strain-hardening effect prevents the necking of the material in accordance with Hart’s criterion. Due to the quite high YS of the SLM material, a lower strain-hardening capability is present. This results in an earlier onset of necking compared with the less work-hardened solution-annealed conventional material, investigated by Choudhary et al. The high scattering of the elongation to fracture at 627 °C may also be a result of this deformation instability.

4.2.2. Influence on the Strength

The DSA is a result of the continuous pinning of dislocations by diffusing atoms. As diffusion is thermally activated, the locking of dislocations occurs only in a certain temperature region, where the diffusion velocity of solute atoms becomes similar to the dislocation velocity. This results in a plateau of the strength over the temperature instead of the typical decrease in strength. Figure 8a shows the YS and the ultimate tensile strength (UTS) of the SLM material in comparison with the data of conventional material available in literature. The SLM material shows a smaller decrease in strength from 300 to 627 °C in comparison with the lower- and higher-temperature regions. However, the data of conventional 316L show even a plateau of the UTS in the DSA-affected temperature range. The SLM material exhibits here a less pronounced strengthening at 627 °C compared with 300 °C due to coarsening of fine substructures and recovery of dislocations with increasing testing temperature.

The DSA mechanism also affects the work-hardening behavior. In contrast to the typical behavior of fcc materials showing continuous softening with increasing temperature, the normalized work-hardening rate of heat-treated SLM 316L material increases between 300 and 627 °C, as shown in Figure 8b, for tensile and compressive loading. The work-hardening rate during compression is higher for tensile loading (at room temperature: 56%), which is possibly due to friction between the stamp and the sample. However, the compression tests reveal that the normalized work-hardening rate of the SLM material is lower at 677 and 777 °C compared with the conventional material as investigated by Choudhary et al. In addition, the comparison of the normalized work-hardening rate from compression tests shows that the SLM material exhibits only a low increase in the normalized work-hardening rate in contrast to the huge increase in the conventional material. This lower work-hardening potential of the SLM material at higher temperatures could be explained by the higher dislocation density and the already well-developed dislocation structure in the SLM material before deformation. Instead, the conventional material is strengthened by the formation of dislocation substructures, which develop at higher strains at elevated temperatures. With regard to the tensile tests, a modified 316L(N) alloy with a higher amount of nitrogen (0.078 wt%), that was solution annealed at 1100 °C, showed a noticeably higher work-hardening rate in comparison with the tensile tests of the SLM material. This difference can be also attributed to the aforementioned increase in dislocation density and the development of dislocation cells during deformation in the conventional solution-annealed material. This assumption is supported by the study of Kashyap et al. on cold-rolled 316L that also showed work-hardening rates similar to the SLM material due to the higher dislocation density of the rolled material.

The investigation of the serrations in the stress–strain curves of the compression tests revealed in the conventional material
two temperature regions where large yield drops occur: According to Choudhary et al.,\textsuperscript{[25]} the first region at around 300 °C could be attributed to the diffusion of interstitial atoms like C or N and the second temperature region from 500 to 627 °C to the diffusion of substitutional solutes Cr and Ni. In contrast to that behavior, no serrations are observed in the first temperature region of the additively manufactured material. However, the serrations in the second temperature region are more pronounced. Also, the increase in the normalized work-hardening rate is less pronounced and slightly shifted to higher temperatures. The reason of this different behavior of the heat-treated SLM material could be due to a generally higher dislocation density. Thus, more diffusing atoms might be needed to lock the large number of dislocations. Moreover, a slight difference in the chemical composition of the investigated alloys regarding the C and N content might be another reason for the different DSA behavior in the lower-temperature regime. Regarding the ductility loss, almost no differences could be observed in comparison with the data on solution-annealed conventional 316L.\textsuperscript{[25]}

The determination of an exact temperature region of the DSA effect in 316L is difficult and it varies also in the literature with onsets from around 200 to 300 °C and an upper limit from around 600 to 650 °C.\textsuperscript{[25–26,54]} The less pronounced work hardening and the absence of large serrations at a temperature of 300 °C lead to the assumption that the DSA effect is less pronounced in the first temperature region in the additively manufactured material. Literature\textsuperscript{[25,56,59]} often mentions the observation of a critical strain for the onset of serrations, but neither the serrations in tensile, nor in compression tests started after an incubation strain. Pham and Holdsworth\textsuperscript{[60]} assume that serrated yielding is pronounced due to vacancy diffusion under tension loading. However, this could not be proved, because no serrations were detected under tensile loading at a temperature of 300 °C and at a strain rate of 10\textsuperscript{−3} s\textsuperscript{−1}.

5. Summary and Conclusion

The present work investigated the microstructure and the mechanical properties of heat-treated SLM 316L material and its influence on the DSA behavior. The following results could be drawn. 1) Heat-treated, SLM-processed 316L exhibits fine substructures combined with a higher dislocation density compared with conventionally produced 316L. EBSD investigations revealed a <101> texture along the building direction. The grain size distribution of the SLM material is wider than that of conventionally produced 316L. 2) The compression strength of the SLM 316L is higher than that of conventionally produced 316L at temperatures up to 877 °C. The heat-treated SLM material is strengthened by the residual substructures. 3) Large drops in the stress–strain curve, which are caused by the PLC effect, were observed in the SLM material only between 500 and 627 °C. The conventional material is additionally affected by serrations in a further low-temperature region at around 300 °C. 4) A negative SRS, an increase in the normalized work-hardening rate, and a smaller decrease in the strength in the temperature range between 300 and 627 °C indicate the DSA to be responsible for the loss of ductility.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

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