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Characterization of Work Hardening Behavior of Additively Manufactured Stainless Steel 316L (1.4404) Using Bulk Metal Forming at Elevated Temperature

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Abstract

A current trend in industry is towards individualized parts with high strength. Regarding geometrical flexibility laser-based additive manufacturing processes like laser powder bed fusion (L-PBF) are applicable. Because of its good weldability the stainless steel 316L is widely used in additive manufacturing (AM) processes. However, the material strength is relatively low compared to other steels. A new approach is the application of a forming operation after additive manufacturing to increase strength due to work hardening. Thus, a fundamental understanding of the forming behavior is crucial to enable a proper process design. Therefore, in this work an upsetting test is used to influence the strength. The aim is to investigate the impact of forming on the mechanical properties. To gain a fundamental understanding of different operating conditions, the forming operation is conducted between room temperature and elevated temperature for conventionally and additively manufactured material. Beside the forming temperature different orientations of the specimens relative to the build direction are taken into account. Finally, the increase of hardness is evaluated and it is shown that the properties strongly depend on the forming temperature and the orientation of the parts during the additive manufacturing process.

Keywords: Additive Manufacturing; Laser Powder Bed Fusion; Stainless Steel; Forming

1. Introduction

In industry a current trend is towards increasing variety of parts and the manufacturing of individualized products, which are adapted to customer demands (Tseng et al., 2014). Therefore, flexible manufacturing process chains are needed to cope with the tendency of quick changes in product development. Within this context the technology of additive manufacturing (AM) is of increasing interest for production processes (Wohlers et al., 2017). Additive manufacturing offers the opportunity of a high degree of geometric freedom and therefore the possibility to manufacture individualized products. One of the most important additive

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manufacturing processes is laser powder bed fusion (L-PBF). In this process, the additive manufactured part is built up layer wise on a substrate plate. The layer wise building of parts enables the process to manufacture individual parts. However, the production times are long compared to conventional manufacturing processes and the material properties depend on a variety of factors like the conditions during the building process and the process parameters. Hence, the resulting material properties of the workpiece underlie deviations, which result from the manufacturing process. In order to improve the mechanical properties post processes like heat treatment or hot isostatic pressing (HIP), a process under high pressure and temperature, are applied to additively manufactured material (Riemer et al., 2014). A promising approach is the combination of additive manufacturing and forming (Schaub et al. 2014). By the application of a forming process the material strength can be increased due to work hardening. However, for a proper process design a fundamental understanding of the forming behavior is crucial. For additive manufacturing processes, the stainless steel 316L, also called 1.4404, is widely used because of its good weldability. Besides, the material offers further beneficial properties like high corrosion and chemical resistance. Therefore, promising fields of application are the automotive sector, chemical industry and medical products (Pramanik et al., 2015).

2. State of the Art

The austenitic stainless steel alloy 316L is developed for parts with high corrosion resistance at room and moderate temperature and consists of a face centered cubic microstructure. Compared to other steels the materials' strength is relatively low. However the strength can be increased by cold-working. For conventionally manufactured material it has to be considered, that for temperatures above approximately 500 °C, the effects of strengthening decrease due to over aging, recrystallization and tempering reactions. (Peckner and Bernstein, 1977) The alloy 316L has lower carbon content than the alloy 316. This improves the weldability, which is beneficial for laser based additive manufacturing processes like L-PBF (Bevan, 2016).

The mechanical properties of additively manufactured 316L are investigated in literature with different focus, since the properties are different to conventionally manufactured material. It is known that the material properties underlie anisotropic behavior because of the layer wise building process, which also includes effects of inhomogeneous metallographic structure (Hitzler et al., 2017). One of the strongest factors of impact is the orientation of the specimen relative to the substrate plate (Sehrt et al., 2009). However, the mechanical properties also depend on several other individual process settings (Sehrt 2010). For example the mechanical properties are influenced by the manufacturing process of the part. By comparing the properties after additive manufacturing with specimens form continuous casting it can be observed that the resulting microstructure and the mechanical properties, characterized by tensile tests, strongly depend on the manufacturing process. The yield strength resulting from tensile tests can be higher than for continuous casting material; however this depends on the orientation of specimen relative to the build direction. It is assumed that the anisotropic behavior is caused by the elongation of grains and orientation of the deposited layers (Gläßner et al. 2017). The orientation of the specimens also influences the fatigue crack growth on compact tension specimens (Fergani et al., 2017). A further factor of impact on the mechanical properties is the porosity of specimens made by additive manufacturing. Therefore, one approach to reduce the pores size is the application of a HIP process after additive manufacturing (Fröml et al. 2018). This can lead to an increase of the ductility compared to AM parts without subsequent HIP process (Leuders et al., 2014).

With regard to prior investigations the mechanical properties of AM parts depend on a variety of factors, but can be improved by post processes. Therefore, the impact of a forming operation on the mechanical properties should be investigated to use the possibility of work hardening and increase the materials hardness for improved mechanical properties. The aim of this work is to evaluate the impact of forming under compression load on the mechanical properties of additively manufactured 316L. In this context the formability is investigated at room temperature and elevated temperature by upsetting tests and subsequently the hardness distribution of the specimens is analyzed. In order to take the orientation of the additively manufactured specimens relative to the build direction into account the specimens are oriented horizontally, vertically and 45 ° to the build plate surface. For comparison the tests are also conducted with conventionally manufactured 316L material, which represents the reference condition to evaluate the impact of the forming operation.

3. Experimental Setup

3.1. Additive Manufacturing

For the L-PBF of the specimens a LT 30 SLM of the 2nd generation from DMG Mori is used. The machine has a build space of 300 x 300 x 300 mm³ and is equipped with a 600 W fiber laser with a Gaussian beam profile and a minimum beam diameter of approximately 70 µm. The basic principle of the process and the specimen orientation relative to the build direction are shown in Fig. 1.

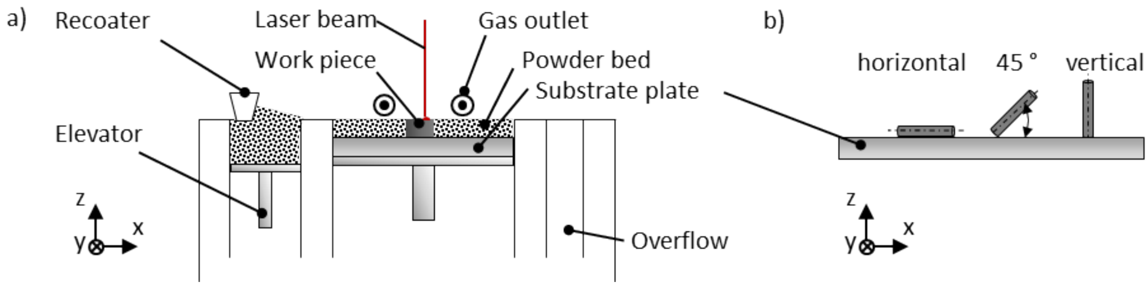

Fig. 1. (a) Principle of the L-PBF process; (b) Specimen orientation relative to build direction

To extract specimens for the subsequent forming operation from the additively manufactured material, cylinders with a length of 70 mm and a diameter of 8 mm are built. For the manufacturing of the specimens a layer thickness of 50 µm and a hatch distance of 0.11 mm are used. The laser power for the hatching process is set to 219 W with a scan speed of 744 mm/s. Since the powder properties can influence the resulting part properties, the powder morphology and the particle size distribution is analyzed by scanning electron microscope (SEM). Based on the measurements generally spherical powder particles are observed. The powder distribution is evaluated by dynamic image analysis (Camsizer X2, Retsch Technologies) and a powder size distribution from 19 µm to 43 µm is measured.

3.2. Forming

In order to evaluate the formability of conventionally and additively manufactured material by upsetting tests, cylindrical specimens with a diameter of 6 mm and the length of 9 mm are used, which are manufactured by turning and grinding from the additively manufactured specimens and a conventionally manufactured semi-finished cylindrical product. The tests allow the characterization of mechanical properties and the investigation of the forming behavior under compression load. The tests at room temperature are conducted with a universal testing machine from the company Walter+Bai with a maximum force of 300 kN. The tool consists of punches made of carbide to handle the high loads during the upsetting test. Teflon film is put between the punch and the specimen to reduce the friction between tool and specimen. The tests are conducted in accordance with DIN 50106 (DIN 50106). The velocity of the punch is set to 5 mm/min and the specimen is compressed to 50 % relative to the initial height. The flow curves are calculated based on the force-displacement-curve. The deflection of the machine is compensated by the control to ensure high accuracy of the test.

In addition to the tests at room temperature, upsetting tests at elevated temperature are conducted. For the tests a thermo-mechanical simulator Gleeble 3500 from the company DSI is used. The principle of the upsetting test and the machine setup is shown in Fig. 2a).

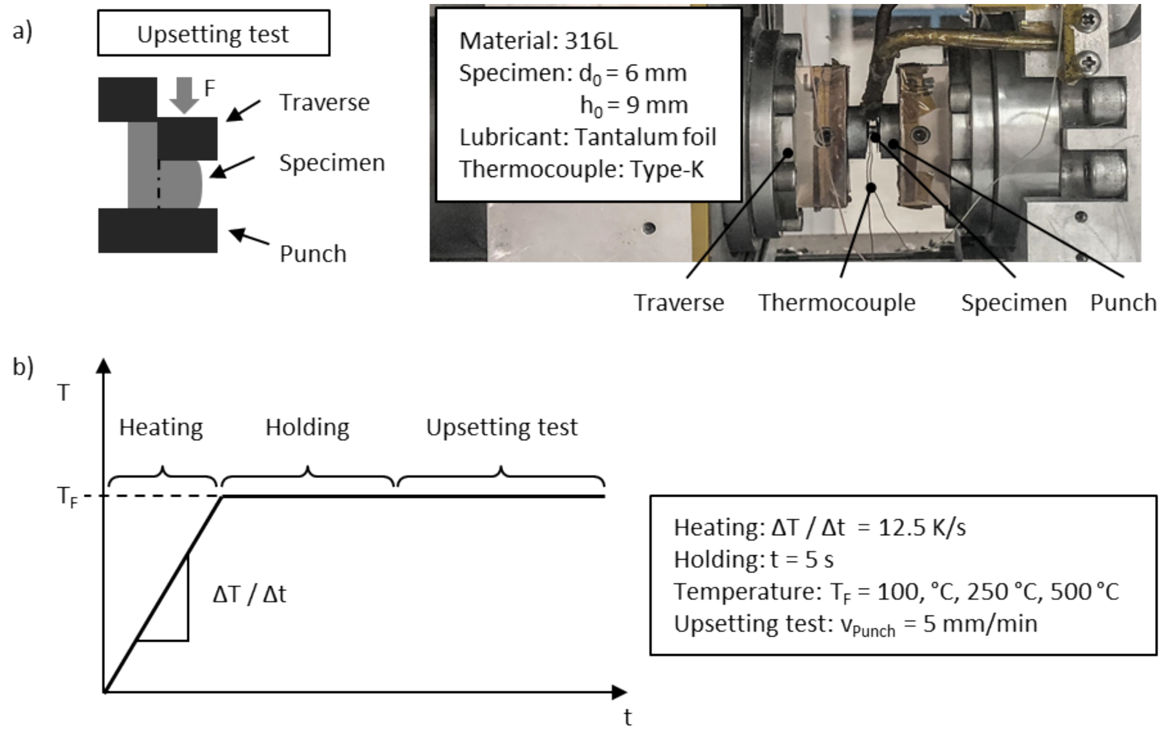

Fig. 2. a) Experimental setup for compression tests at elevated temperature in a Gleeble 3500 b) Time-Temperature-Curve of the tests

The heating process is based on direct resistance heating. The maximum force is limited to 100 kN. With regard to the tests at room temperature the same punch velocity is applied. The displacement of the punch is measured by a linear variable differential transformer (LVDT) to achieve a precise measurement without machine deflection. Since the heating process is realized by resistance heating instead of a Teflon film an electrically conductive tantalum foil is used for the reduction of friction. The temperature of the specimen is measured by thermocouple of type K which is welded on the surface of the specimen. However, the temperature is measured in the center of the specimen. But the resistance heating lead to a temperature gradient from the center to the edge of the specimen. Therefore, the precise forming temperature is reached only in the middle of the specimen. The time temperature profile (Fig. 2b) consists of three parts. The first part represents the heating phase to heat the specimen from room temperature to the target temperature. The average heating rate is set to 12.5 K/s. Afterwards the temperature is held for 5 s to ensure a homogeneous heating of the specimen core to the target temperature, since the temperature is measured on the surface. Finally, the forming operation is conducted and the flow curves are calculated based on the force-displacement data. In order to receive a broad knowledge of the temperature impact, the tests are conducted at 100 °C, 250 °C and 500 °C. Since work hardening should be used to increase hardness and the effect strongly decreases for conventionally manufactured 316L above 500 °C (Peckner and Bernstein, 1977), higher temperatures are not investigated.

3.3. Metallographic analysis

In order to investigate the metallographic structure of conventionally and additively manufactured material cross sections are analyzed before and after the forming operation. The structure before forming represents the initial state and is used as reference to evaluate the change through the forming process. The specimens are polished and subsequently etched with V2A-etchant for approximately 20 s. The analysis is done with a reflected light microscope Aristomet from Leitz.

3.4. Hardness measurement

To evaluate the impact of forming on the work hardening of the additively and conventionally manufactured material, the hardness is measured before and after forming. The hardness measurement is conducted with a Fischerscope HM2000 from Helmut-Fischer GmbH, where the impact force is set to 500 mN. For a comprehensive investigation of the hardness an array of measurement points is used. The distance between the points is 0.3 mm in vertical and horizontal direction. The hardness before the forming operation is measured at 50 points, which represents the reference condition to evaluate the increase of hardness after forming.

4. Results

4.1. Formability

The formability is analyzed for conventionally manufactured material and additively manufactured material for three different orientations; horizontal, vertical and under 45 ° to the build direction. Furthermore, the forming temperature is changed on four stages between room temperature and 500 °C. By using the force displacement data from the machine, the flow curve is calculated, which represents the flow stress over the true plastic strain. The resultant curves for the different combinations are shown in Fig. 3 a-d). For each parameter combination three tests are conducted, therefore the diagrams include representative curves of the tests. In general, a reduction of the flow stress is measured with an increase of temperature. The decrease of flow stress for 100 °C (Fig. 3b) is comparatively low. A distinct reduction of the flow stress can be observed at 250 °C (Fig. 3c) and 500 °C. However, the difference between 250 °C and 500 °C (Fig. 3d) is rather small. The highest differences between the flow curves for additively manufactured material and for the three different orientations of the specimens are present at room temperature. At room temperature (Fig. 3a) the horizontal orientation has the highest stress at the end of test, compared to 45 ° and the vertical orientation. But this difference decreases for increasing temperature (Fig. 3a-d). This shows that the anisotropic behavior of different specimen orientations during the AM process interacts with the forming temperature. For strains below approximately 0.2 the flow stress for conventional manufactured material is higher than for additively manufactured material. This difference remains for all investigated temperatures. Beside the flow behavior during the forming operation, the yield stresses are analyzed (Fig. 4) to quantify the differences between conventionally manufactured material and additively manufactured material as well the impact of the temperature. With regard to the tests at room temperature (Fig. 4), the yield stress of the conventionally manufactured material (525 ± 12 MPa) is almost on the same level than the yield stress of the additively manufactured material in horizontal orientation (536 ± 14 MPa). However, in consideration of to the standard deviation significant differences cannot be identified. Whereas, the yield stress for an orientation of 45 ° is slightly higher (551 ± 9 MPa) than for specimens build in vertical direction (514 ± 9 MPa).

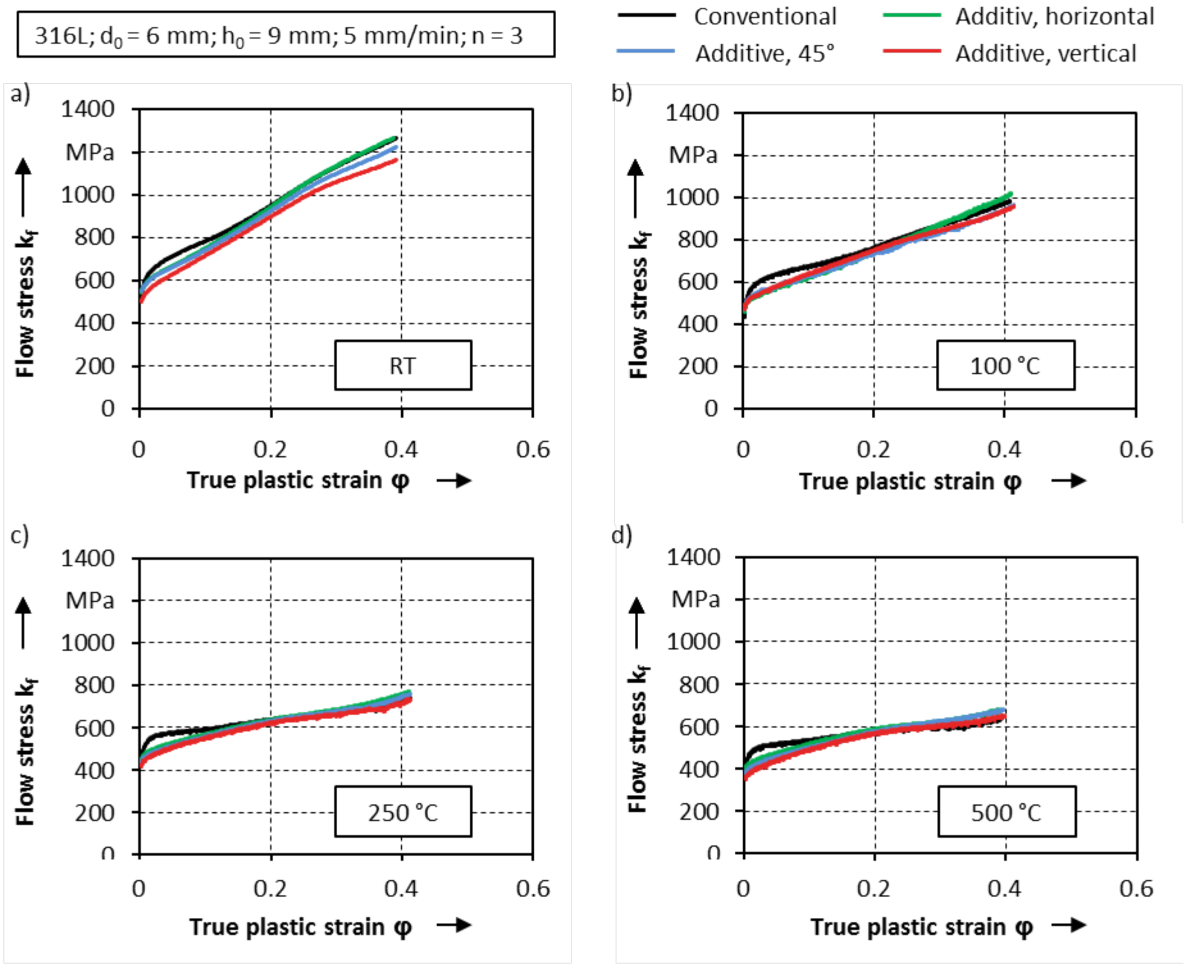

Fig. 3. Flow-curves for a) RT, b) 100 °C, c) 250 °C and d) 500 °C for conventionally and additively manufactured material

This tendency of different yield stresses in dependency of the specimen orientation remains for a temperature of 100 °C, but for forming at 250 °C and 500 °C the difference declines and cannot be identified for the additively manufactured material since the standard deviations overlap. For a temperature of 250 °C it can be observed that the yield stress of the conventionally manufactured material is higher than for the additively manufactured material. The behavior remains for a forming temperature of 500 °C. This indicates that the forming behavior strongly depends on the temperatures but also on the manufacturing process of the material, since the yield stress of conventionally manufactured material is higher than for additively manufactured material for temperatures above 250 °C. In order to link the mechanical properties to the grain alignment due to the different specimen orientations, the metallographic structure is analyzed.

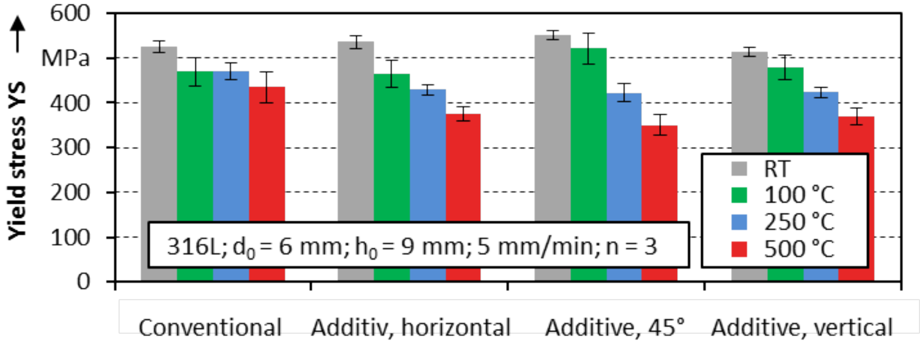

Fig. 4. Initial yield stresses for conventionally and additively manufactured material at different temperatures

4.2. Metallographic structure

For a more in-depth analysis of the forming behavior of different orientations of additively manufactured specimens, the metallographic structure before the forming operation is analyzed. The results for conventionally and additively manufactured material are shown in Fig. 5. The conventionally manufactured material has a small grained structure compared to the additively manufactured material. The AM material is dominated by the shape of welding beads, which result from the single melt pool tracks of the L-PBF process. The welding beads are oriented correspondingly to the build direction. This means that in the case of the horizontal orientation the welding beads are aligned 90 ° to the force direction of the forming operation and diagonal under 45 °.

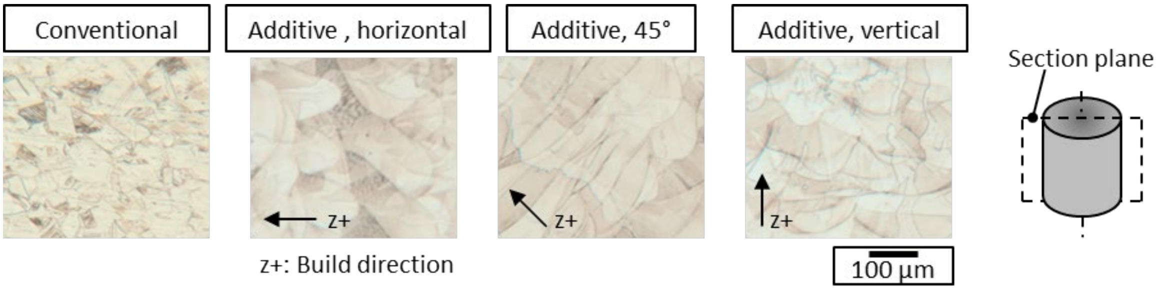

Fig. 5. Metallographic structure before forming for conventionally and additively manufactured materials with different orientations

4.3. Hardness

For a comprehensive evaluation of the mechanical properties, the hardness is measured prior and after the forming process. The initial hardness values before the forming operation are shown in Table 1. With regard to the measured values it can be observed that the hardness values are almost on the same level, which is beneficial to evaluate the impact of forming on the increase of hardness. However, the hardness of specimens with 45 ° orientation is slightly lower than for the specimen in vertical build direction. This result could be reasoned by a temperature distribution in the specimen during the build process, which could be influenced by the different support conditions of the three orientations, since the heat in the specimen is expelled also by the support structure. In consideration of the standard deviation a higher amount of measurement points would be beneficial for further investigation of this phenomenon.

Table 1. Initial hardness before forming

Material / Orientation	Mean value	Standard deviation
Conventional	315,1 HV 0.05	± 21,2 HV 0.05
Additive, horizontal	308,3 HV 0.05	± 25,2 HV 0.05
Additive, 45 °	285,9 HV 0.05	± 17,1 HV 0.05
Additive, vertical	339,2 HV 0.05	± 27,7 HV 0.05

To evaluate the impact of forming on the hardness the specimens are also analyzed after the forming operation. In Fig. 6 are shown the metallographic structure (Fig. 6a) after the forming operation at room temperature and also the corresponding hardness distribution (Fig. 6b).

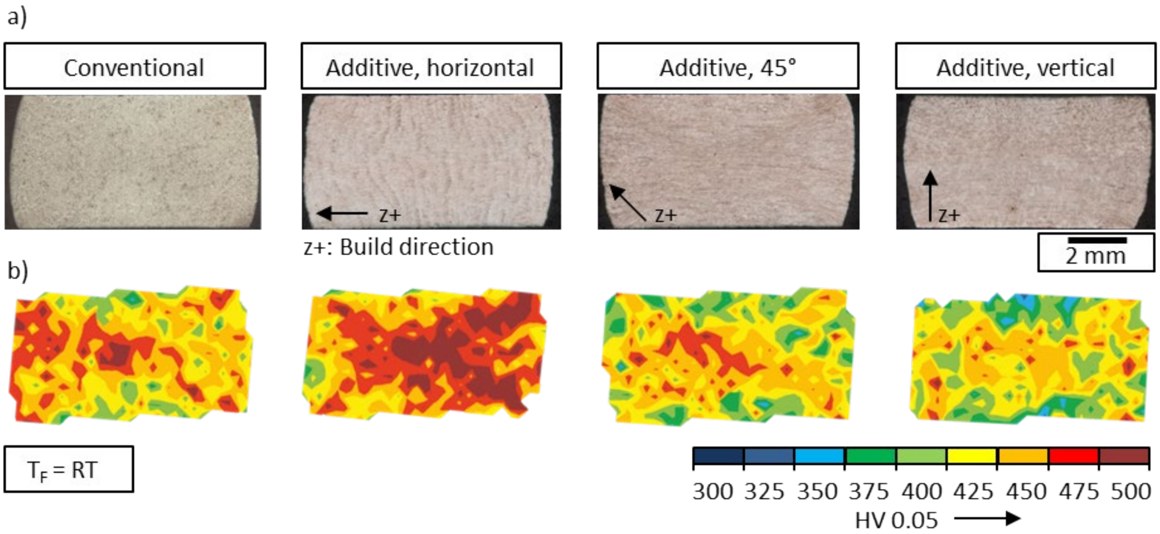

Fig. 6. a) Metallographic structure after forming for conventionally and additively manufactured materials at different orientations
b) Hardness distribution after forming

The conventionally manufactured material shows the typical forming zone after an upsetting test, where the highest compression of the grains is located in the center of the specimen, whereas the grains at the edge are hardly formed. This is indicated by the microsection, where the highly formed area appears darker than the hardly formed area. Hence, the hardness has the highest values in the center of the specimen. When comparing the additively manufactured material, it can be observed, that the increase of hardness strongly depends on the initial orientation of the specimen during the AM process. The highest hardness values are reached for the horizontal orientation. In this case the hardness in the center of the specimen is even higher than for the conventionally manufactured material. In contrast, the hardness for specimens with 45 ° orientation is lower than for the orientation in horizontal direction, but the hardness distribution is comparable to that of the conventionally manufactured material. The lowest hardness values are identified for the additively manufactured specimens with vertical orientation. A possible explanation for the orientation dependent behavior could be the deformation of the grains. In case of the horizontally oriented specimens the longitudinal direction is aligned in forming direction. Therefore the distortion of the grains due to forming under compression load might be higher than of the specimen with 45 ° or vertical direction.

For evaluating the impact of the forming temperature and to identify process conditions to increase the hardness of additively manufactured material, in Fig. 7 are shown the hardness distributions for forming temperatures of 100 °C and 250 °C for conventionally and additively manufactured material in vertical direction. The vertically oriented specimen is of special interest, since this shows the biggest difference in hardness after forming compared to conventionally manufactured material. The hardness of the conventionally manufactured material at 100 °C is lower than at room temperature, which corresponds with the effect of decreasing work hardening with increasing temperature. However, at 250 °C the resulting hardness is much lower compared to forming at room temperature or 100 °C. In general a similar behavior can be observed for additively manufactured material in vertical direction. The hardness is much higher at a forming temperature of 100 °C compared to 250 °C. With regard to conventionally manufactured material the hardness seems to be higher at 100 °C for the AM material, even though the specimens with vertical orientation showed a lower hardness after forming at room temperature. This shows that the hardening behavior depends on the forming temperature and can be influenced by the process parameters. Whereas the hardness after forming at 250 °C lead to a similar result for conventionally and additively manufactured material. The differences in hardness indicate changes in the microstructure due to forming and temperature. Therefore, the metallurgical processes will be analyzed in detail for the investigated temperature range until 500 °C. In this context effects of blue brittleness (Bargel, 2018) and thermal induced precipitations have to be taken into account.

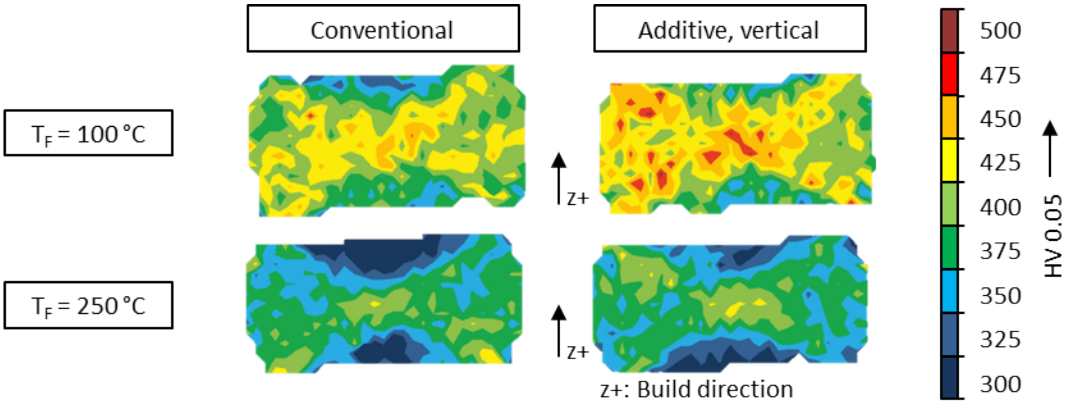

Fig. 7. Hardness distribution for conventionally and additively manufactured material at 100 °C and 250 °C

5. Summary and outlook

In this work a forming operation after additive manufacturing is applied to improve the mechanical properties of 316L. It is investigated how the hardness can be influenced by the forming operation and to show the difference of additively manufactured material the tests are conducted also for conventionally manufactured material. The formability of conventionally and additively manufactured material 316L is analyzed by upsetting tests at room temperature and elevated temperature, which are 100 °C, 250 °C and

500 °C. In order to analyze anisotropic effects of additively manufactured material the orientation of the specimen relative to the build direction is varied. The specimens are built in three different orientations; vertical, horizontal and 45 ° to the build plate. Finally the impact of the forming operation on the increase of hardness is evaluated to investigate proper process parameters to influence the work hardening behavior.

The findings of this work show that the increase of hardness can be influenced by the forming temperature. Forming at room temperature results in the highest hardness values. However, forming above 250 °C decreases the hardening of the material in order to the forming operation. Furthermore, the increase of hardness depends on the orientation of the specimens relative to the build direction. The highest hardness values are reached for specimens in horizontal orientation.

These results are based on the investigated temperatures. Hence, future work will be focused on forming at further temperatures to receive broad process knowledge about forming to influence the mechanical properties of additively manufactured material.

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