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Experimental investigation of mechanical properties of the AISI 316L stainless steel: macro- and microscale

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Abstract
Additive manufacturing (AM) has recently gained popularity over the conventional manufacturing methods as it allows for innovative design solutions in terms of geometric complexity and shape optimization. As a result of the geometry, size and heterogeneous microstructure of the AM samples, conventional macroscale experiments are, however, frequently inadequate in determining the resulting material parameters. In such cases, nanoindentation represents a viable non-destructive technique for determining the mechanical properties such as Young's modulus (E_{IT}) and hardness (H_{IT}) of small samples' volumes. The nanoindentation measurements on a Keysight G200 machine equipped with a standard Berkovich tip are performed in this study on AISI 316L wrought samples as well as samples obtained via the laser-powder bed fusion (L-PBF) AM process. The standard depth-controlled loading-unloading method is used. With the aim of comparing the material properties on the macro- and microscale, conventional monotonic uniaxial tensile tests are also performed on both samples types. The possibility of calibrating the material parameters of finite element (FE) numerical models using nanoindentation test results to obtain the load vs. the indentation depth curve is finally discussed. Experimentally obtained load-displacement curves are used to calibrate the elastic and plastic material parameters of the FE model. Since FE modelling of the nanoindentation process is a nonlinear elasto-plastic contact problem, Young's modulus and hardness alone are, in fact, not sufficient to characterize properly the overall material behaviour.

Additive manufacturing, L-PBF, AISI 316L, nanoindentation, monotonic uniaxial tensile test

1. Introduction

Additive manufacturing (AM) offers many advantages over conventional manufacturing methods. It has, therefore, been sparking a growing interest, as it permits new design solutions in terms of geometric complexity and shape optimization, together with rapid product development and sustainability. Three-dimensional (3D) metallic parts can be produced via the AM process directly from computer aided design (CAD) models without the use of a traditional tooling machine. One of the most promising AM techniques is laser-powder bed fusion (L-PBF), consisting of layers of atomized metal powder that are selectively melted by a laser beam to create the final 3D geometry [1]. The mechanical properties of parts produced by using the L-PBF method are influenced quite a lot by the AM process parameters. There is then a rich body of recent literature indicating that the mechanical properties resulting from AM processes are enhanced with respect to those of conventionally manufactured materials in terms of e.g. tensile and yield strengths, corrosion and wear properties and fatigue lifetime [2]. An accurate mechanical characterization is thus important for the proper design and development of new components.

Nanoindentation as a non-destructive technique can be used as a method to thoroughly quantify the mechanical properties of AM samples. In fact, the nanoindentation technique is increasingly being used for the mechanical characterization of structural metals in terms of hardness and modulus of elasticity. Such an approach enables also significant costs reductions compared to the conventional macroscale methods, since much smaller sample volumes are sufficient. During the nanoindentation tests, the material typically experiences an elasto-plastic deformation during loading, followed by an elastic recovery during unloading. Although the nanoindentation technique is well established for determining elastic properties, its employment for quantifying the plastic properties is not yet clear. To estimate the plastic properties, an inverse analysis combining nanoindentation tests and a detailed elasto-plastic finite element (FE) model could, therefore, be a useful approach [3].

Due to its excellent resistance to corrosion, mechanical strength and ductility, the AISI 316L stainless steel is considered one of the attractive metallic materials in many industries, such as food, mining, petrochemical, construction or shipbuilding. Over the years, AISI 316L has also gained popularity in medicine, in particular in surgery and dentistry [4, 5].

Several investigations studying separately only the macro- [6, 7] or only the micro- [2] mechanical properties of AISI 316L produced via the L-PBF AM process have been carried out so far. Other studies have concentrated separately on conventionally and on materials produced by L-PBF. This work, in turn, investigates experimentally and compares the mechanical properties at the macro- and microscale obtained for additively and traditionally produced AISI 316L steel samples. In an effort to reduce the number of tests, an attempt to provide an insight into the development of advanced FE models is also made.

2. Materials and methods

The tested materials are wrought and L-PBF AM produced AISI 316L stainless steel with chemical composition and process parameters reported in [7, 8]. The AM specimens are

manufactured as cylindrical rods of 180 mm height and 23 mm diameter (Fig. 1).

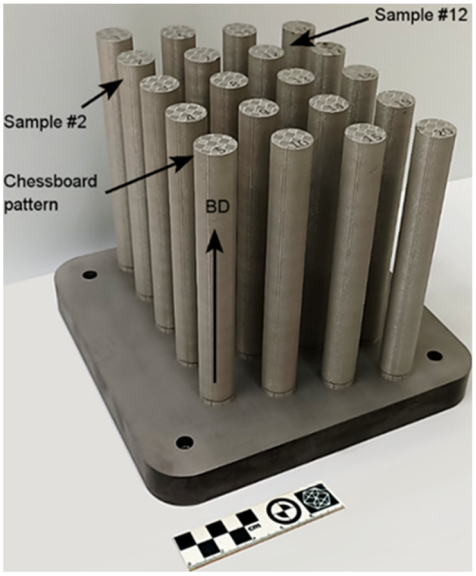

Figure 1. Specimens produced by the AM L-PBF technique and respective build direction (BD) [7]

Each specimen is then turned to achieve the final cylindrical dog-bone specimen geometry (Fig. 2).

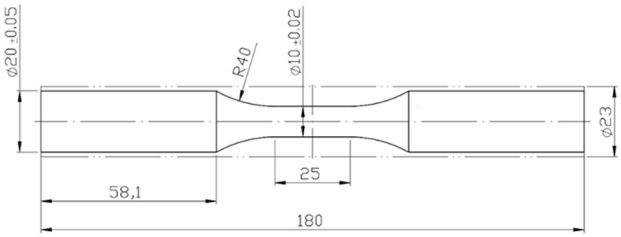

Figure 2. Specimen geometry turned from vertical cylindrical rods (dash double-dot lines) to dog-bone geometry (solid lines) [7]

2.1. Monotonic uniaxial tensile tests

Monotonic uniaxial tensile tests were carried out first on cylindrical specimens with uniform gauge sections of 25 mm in length and 10 mm in diameter (as per ASTM E606, Fig. 2), while using the experimental set-up shown in Fig. 3 thoroughly described in [7, 8].

Figure 3. Monotonic uniaxial tensile test of wrought AISI 316L

2.2. Nanoindentation experimental tests

The nanoindentation tests are conducted on a Keysight G200 nanoindenter (Fig. 4), which has a load resolution of 50 nN and a displacement resolution of 0.01 nm. A standard loading-unloading method is used in all experiments, while the temperature in the measurement chamber is kept constant at 24 °C.

Two samples are extracted herein from the rods: one produced via AM and the other as wrought material. The samples are then embedded in an epoxy resin and ground and polished to obtain an optical surface quality. Six different indentation depths (h_{max}) are considered: 500 nm, 1000 nm, 1500 nm, 2000 nm, 2500 nm and 3000 nm, and nine indentations are performed in both materials at each indentation depth. Nanoindentation Young's modulus (E_{IT}) and hardness (H_{IT}) values are determined for each test independently.

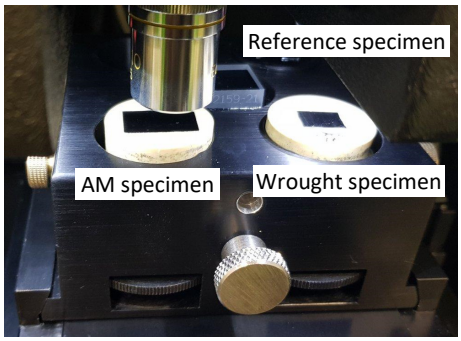

Figure 4. Specimens positioned in the Keysight G200 nanoindenter sample holder

In Fig. 5 is depicted a typical experimentally obtained load-displacement (*P-h*) curve in which P_{max} represents the maximal indentation load, h_{el} is the elastic recovery, and h_{pl} indicates the residual (plastically deformed) depth after a complete unloading cycle.

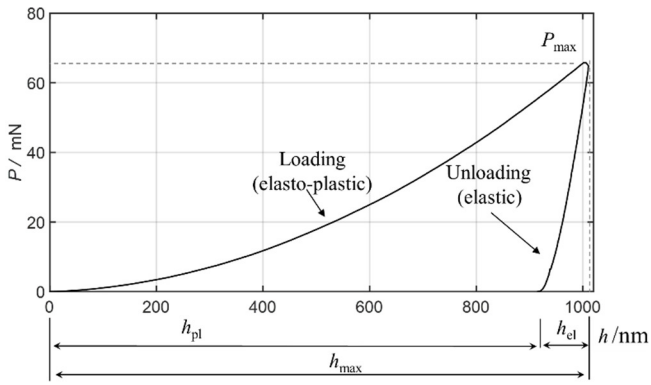

Figure 5. Typical load-displacement curve obtained experimentally at h_{max} = 1000 nm

2.3. Numerical simulations

The nanoindentation tests are simulated by using the finite element analysis (FEA) implemented in the Ansys® commercial computer code. The experiment is modelled numerically as a nonlinear two-dimensional (2D) axisymmetric FE problem, where contact itself is defined as a sliding surface without friction. Since nanoindentation is a contact problem, a very fine mesh between the indenter and the specimen is required to resolve the large stress and strain gradients and to impose accurately the contact elements. To minimize the computational time, the mesh is refined at the indentation site and made

coarser away from it, resulting in a total of 14 628 three-node triangular elements with 4 949 nodes (Fig. 6). The symmetry boundary condition is applied along the *y* axis, while the lower edge of the model is assumed to be fixed. The Berkovich indenter is modelled as a rigid "line", and displacement-controlled loading is imposed on its tip in all simulations. The geometrical parameters of the numerical model are chosen based on the results presented in [9] so as to minimise their influence on the obtained results. The material response of the samples is assumed to be bilinear elasto-plastic.

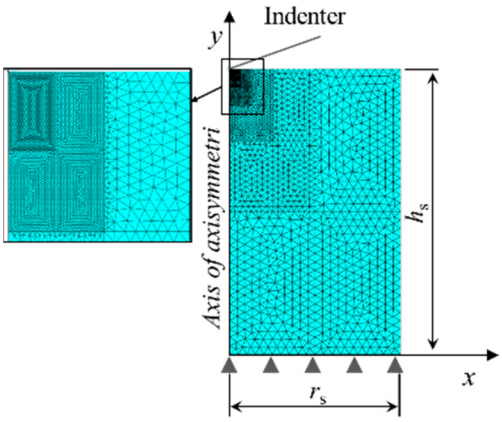

Figure 6. Axisymmetric FE model with significant constraints and geometrical parameters [9]

The ultimate goal is to develop a methodological procedure that can provide a set of unique elasto-plastic material parameters based on the experimental *P-h* curves. Young's modulus and hardness are herein the known values, experimentally obtained from nanoindentation tests, while the parameters such as yield stress (Y) and tangent modulus (E_t), characterising the plastic behaviour of the adopted bilinear elasto-plastic model, are the unknown values.

3. Results and discussion

The results of the performed tensile tests conducted to assess the mechanical properties of wrought and L-PBF AISI 316L stainless steel samples [7, 8] are compared to those obtained via nanoindentation experiments. Table 1 shows the elasto-plastic material parameters, i.e., Young's modulus (E), yield stress ($Y_{0.2\%}$) and tangent modulus (E_t), obtained from the monotonic uniaxial tensile tests.

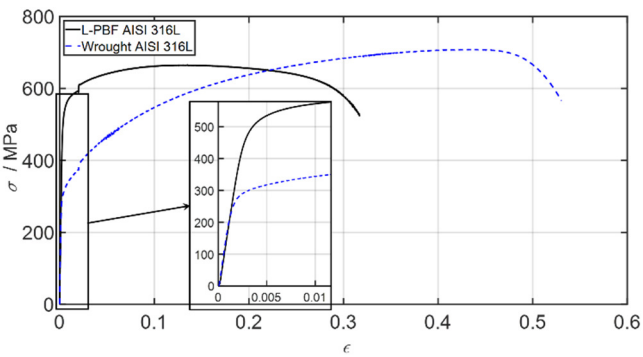

Figure 7. Experimentally obtained monotonic uniaxial tensile curves for wrought and L-PBF AISI 316L

In Fig. 7 are shown the respective curves obtained for both types of the AISI 316 stainless steel samples. The usual offset of 0.2% is adopted to evaluate the yield stress for both materials.

Base on the data in the Figure 7 and in Table 1 it can be noted that, although both materials exhibit quite a similar behaviour up to the elastic limit, i.e., the Young's modulus values differ by ~ 1 % only, the value of the yield stress is ~ 40 % higher in case of the AM-obtained material. These results are consistent with those reported in previous art [2, 10].

Table 1. Material properties obtained from the monotonic uniaxial tensile test for wrought and L-PBF AISI 316L stainless steel

Material	E/GPa	$Y_{0.2\%}$/MPa	E_T/MPa
Wrought	197	305	4989
L-PBF	199.3	529	5499

Table 2 summarizes, in turn, the nanoindentation Young's modulus (E_{IT}) and hardness (H_{IT}) values calculated for each indentation depth. As it can be seen from the reported results, the uniaxial tests produce roughly 4 % higher E values compared to the equivalent E_{IT} (averaged for all indentation depths) for both types of the AISI 316L specimens.

Table 2. Young's modulus and (E_{IT}) and hardness (H_{IT}) values for different nanoindentation depths

	Wrought		*Additive*	
h_{max}/nm	E_{IT}/GPa	H_{IT}/GPa	E_{IT}/GPa	H_{IT}/GPa
500	186.6	3.06	192.1	3.14
1000	192.5	2.74	192.6	3.06
1500	188.1	2.53	185.9	2.81
2000	189.5	2.49	194.7	2.94
2500	190.3	2.55	189.6	2.76
3000	187.1	2.45	189	2.73
Average	189.02	2.64	190.6	2.91

In Fig. 8 are compared the nanoindentation tests on both considered materials and all indentation depths. It can be observed that in the case of additively manufactured specimens higher indentation loads are needed to achieve the set nanoindentation depth. What is more, the difference between the two material types is more pronounced as the indentation depth increases.

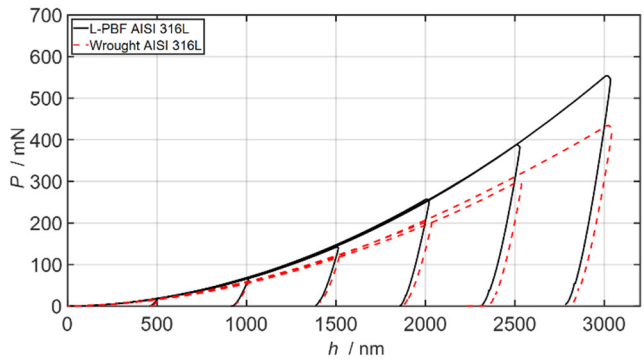

Figure 8. Load-displacement curves obtained experimentally for L-PBF and wrought AISI 316L stainless steel samples at different indentation depths

In an attempt to develop a methodological procedure that could provide unique elasto-plastic material parameters based on the obtained nanoindentation experimental results

presented above, several runs of numerical sensitivity analyses are performed in the next step of the work. Two different sensitivity analyses (marked as sets A and B) are therefore performed with the ratios Y/E_t varying from 0.02 to 0.25. In the first set (A), the value of the tangent modulus E_t is kept constant (10 000 MPa), while the values of the yield stress Y are varied from 200 to 2500 MPa. In set B, the yield stress is kept constant (600 MPa), while the tangent modulus is varied from 2400 to 30 000 MPa. All the numerical simulations are then performed considering a nanoindentation depth of 2000 nm.

To better understand the obtained results, the loading and unloading parts of the *P-h* curve are considered separately. The loading part is thus used to monitor the maximum load, while the unloading part gives an indication about permanent deformations.

In case of the loading curves, the sensitivity analyses showed that by increasing the Y/E_t ration, the maximum load increases for set A, with a 70.5 % relative difference between the first and the last test. On the other hand, set B showed that the maximum load decreases with increasing Y/E_t values, with a relative difference of 59 %.

When considering the unloading curve, an increase of Y/E_t gives rise to an increase of the permanent deformation of, respectively, ~ 200 % for the parameters' set A, and of ~ 318 % for set B.

Finally, two sets of relations are defined based on the observed trends of Y/E_t vs. h_{pl} and Y/E_t vs. h_{el} for the sensitivity analyses sets A and B. The obtained relations are then used to determine the elasto-plastic parameters (Y/E_t), where the experimentally obtained elastic and plastic deformation parameters are used as input data.

In Fig. 9 are presented the obtained results, depicting the comparison of experimental and numerical *P-h* curves for a nanoindentation depth of 1000 nm. It can be noted that, with respect to the experimental results, the numerically obtained curve shows a difference in the maximum load of 3.2 % and underestimates the permanent deformation by 10.5 %. These results are rather encouraging but, bearing in mind also the complexity of the analysed nonlinear contact problem, further analyses are required to converge to the aimed methodological procedure enabling the determination of a unique set of values of the elasto-plastic parameters that would be valid for different materials. This challenging task remains part of our ongoing work.

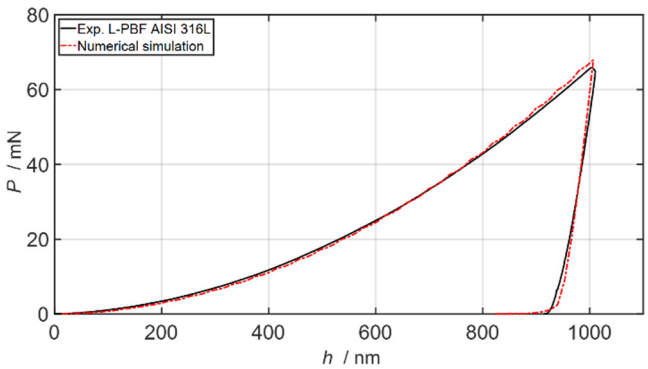

Figure 9. Comparison between experimental and numerical *P-h* curves for a 1000 nm indentation depth on the L-PBF AISI 316L stainless steel sample

4. Conclusions and outlook

The research in this paper examines and compares the mechanical properties of the AISI 316L stainless steel obtained via conventional manufacturing and by using the additive manufacturing laser-powder bed fusion technique. Monotonic uniaxial tensile tests are performed first on an experimental apparatus described in [7, 8]. These experiments are followed by depth-controlled loading-unloading nanoindentation tests performed on a Keysight G200 nanoindentation system using a Berkovich tip. The obtained experimental results allow determining that the uniaxial tests produce approximately 4 % higher Young's moduli with respect to the indentation values averaged over all considered indentation depths. What is more, in the case of uniaxial tests both materials have similar Young's moduli, while the value of the yield stress is approximately 40 % higher for the AM sample.

Using the nanoindentation test results, an attempt to develop a methodological procedure that could provide a unique set of values of the elasto-plastic material parameters, two initial sets of sensitivity analyses are performed. The load-displacement curve obtained experimentally is used to calibrate the elastic and plastic material parameters, and initial encouraging results are obtained. In future work these results will be further refined in order to obtain an improved model that can accurately simulate the behaviour of different materials.

Acknowledgements

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