FABRICATION PROCESS AND BASIC MATERIAL PROPERTIES OF THE BASF ULTRAFUSE 316LX MATERIAL

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The presented article deals with the research of commercially available BASF Ultrafuse 316LX filament intended for processing by the Fused Filament Fabrication additive technology. This material contains a high proportion of metal particles. The aim of the research was to compare the resulting mechanical properties of the Ultrafuse 316LX not only with conventional rolled AISI 316L stainless steel, but also with the AISI 316L material processed by another additive technology – Selective Laser Melting. Several sets of tensile test specimens were printed from Ultrafuse 316LX using Felix Tec4 machine to determine specific mechanical properties. The same sets of samples were made from AISI 316L powder using Selective Laser Melting additive technology for direct comparison of selected mechanical properties. After the whole manufacturing process, the Ultrafuse 316LX show very interesting mechanical properties with adequate strength and increased ductility.

KEYWORDS

Additive manufacturing, Fused Filament Fabrication, FFF, Metal printing, Mechanical properties, SEM, sintering.

INTRODUCTION

3D printing is a progressive fabrication method that allows the production of parts with very complex shape. This is ensured by layer-by-layer adding of the material [Chua 2014]. Using this approach, it is possible to lighten inner core of the part and thus save the input material [Wohlers 2014]. Nowadays, the 3D additive technologies enable to process various materials such as metals, ceramics and photopolymers. [Gibson 2014].

Currently, metal 3D printing is most often realised with the use of so-called powder bed technologies. Main defining representatives are Selective Laser Melting (SLM), Direct Metal Laser Sintering (DMLS) and Electron Beam Melting (EBM) [Buchanan 2019]. In the frame of the printing process, the material is melted in the inert atmosphere or vacuum. However, there are other technologies on the market, such as BinderJet, which uses special binders (adhesives) to join the individual grains of the processed metal material. However, the resulting model must undergo other processing steps that ensure its resulting mechanical properties. The main disadvantage of the technologies described above is their high acquisition and running costs. For this reason, BASF decided to use its knowledge of Metal Injection Moulding (MIM) technology and transfer it into the field of 3D printing, specifically FFF technology. The result of their research activities is the Ultrafuse 316LX material, which contains more than 80 percent of 316L metal powder [Forward AM 2020]. This metal powder is supplemented with a polymeric carrier, which allows the material to be extruded into the form of a filament, which can be further processed by FFF technology. At the current level of knowledge, only few articles deal with production of Ultrafuse 316LX material on standard FFF machines [Thompson 2019]. Therefore, this article tends to fill this gap by mapping the fabrication process on the commercially available Felix Tec4 printer and by evaluation of basic mechanical properties of the material.

FFF technology is widely used for 3D printing of polymeric materials – plastics [Fernandez 2015]. Currently, there are a large number of manufacturers of machines for 3D printing of plastics. On the other hand, development of equipment dedicated to FFF 3D printing of metals is not solved in such a wide range. There are now two major players in the commercial sector which process metal powder in the form of the filament. Markforged, Inc. is the first one with Atomic Diffusion Additive Manufacturing (ADAM) technology and Metal X equipment [Markforged 2020]. Desktop Metal, Inc. is the second, equally important player with the Studio System printing solution [Desktopmetal 2020]. The first mentioned Markforged uses metal in a polymer carrier in the form of a long string (filament) wound on a spool. The Desktop Metal uses metal in a special wax polymer, which is provided in the form of short, narrow rods. Both companies use two types of materials during their printing process. The first material forms the product body and also the support structures. Second material consists of a ceramic powder and a carrier and it is intended as a separation site between support structures and the part. With this approach, the part and support structure are prevented to merge together during further chemical and thermal processes.

BASF ULTRAFUSE 316LX FILAMENT

Ultrafuse 316LX material is a product of BASF 3D Printing Solutions GmbH. It is a material intended for processing on any 3D printer designed for FDM or FFF technology. The material consists of a polymeric carrier (binder) and AISI 316L (DIN 1.4404) stainless steel metal powder. Figure 1 shows a cross-section of the filament under scanning electron microscope (SEM).

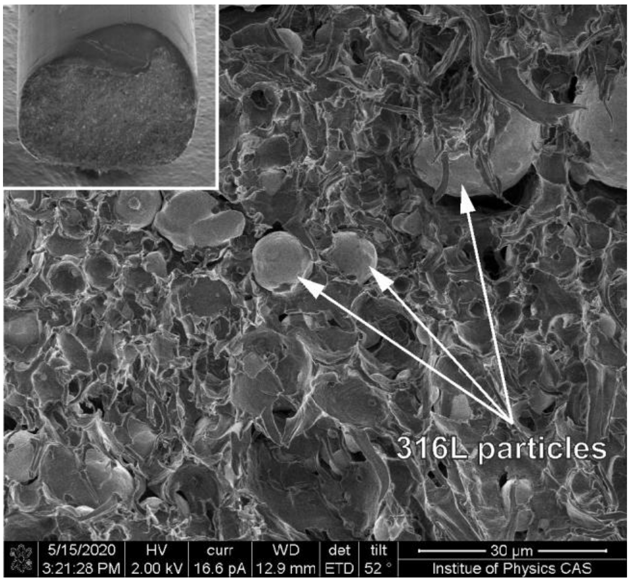

Figure 1: SEM images of BASF Ultrafuse 316LX filament cross-section

BASF indicates metal powder content within the filament of more than 80 wt %. BASF developed Ultrafuse 316LX based on its experience with Metal Injection Moulding (MIM) technology [Gong 2018]. The material is thus based on materials dedicated for MIM technology, where BASF has been operating as a supplier of materials for many years. The procedure for working with the Ultrafuse 316LX material is thus identical with the procedure for processing metal products using MIM technology. The 316L particles in the filament were examined using Energy-dispersive X-ray spectroscopy (EDS) for evaluation of its chemical composition. Results are summarized in Table 1. The composition is in agreement with standard specifications of AISI 316L stainless steel [AZOM 2020].

Element	Wt. %	At. %
Si	0.9	1.63
Mo	2.98	1.59
Cr	17.07	16.9
Fe	66.3	60.78
Ni	9.29	8.1

Table 1. Chemical elements of at the filament ULTRAFUSE 316 LX sample.

In the first step, the so-called Green Part is made. It is a part containing a polymeric carrier with a metal. The Green Part must then go through the debinding process, where most of the binder is removed and the model enters its next phase, the so-called Brown part. The brown part already composes almost exclusively of metal powder with only a minimal amount of binder on the edges of the metal powder grains and it does not yet achieve the required mechanical properties. The brown part is therefore very prone to damage of the geometry during manipulation.

In the last step, the part is sintered, thus removing the residual binder and joining the metal particles together. After this operation, the final metal part without any internal stress is ready. During sintering, the parts shrink. Excessive shrinkage can also lead to deformations of the geometry, if the part or its support is improperly designed. The entire process of Ultrafuse 316LX filament preparation together with its processing by FFF technology and post-processing operations including debinding and sintering of the part is shown in Figure 2.

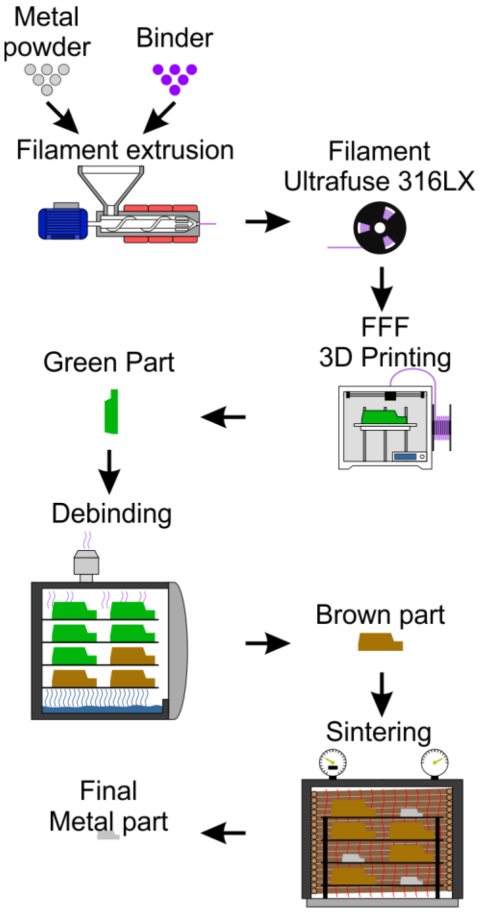

Figure 2. Technological process for producing the final part from BASF 316LX filament

MATERIALS AND METHODS

Mechanical testing and subsequent analysis of gathered data offer the possibility to compare different groups of tested materials. For the purpose of this paper, tensile test was chosen for qualitative assessment of the basic mechanical constants.

1.1 Fabrication of specimens

Here performed tensile tests were not done in accordance with usual standards for metallic materials such as ISO 6892 and ASTM E8. This is due to the fact that these standards recommend specimens with circular cross-area, typically with the diameter of 6 mm. With respect to current technological limitations of FFF technology for processing of metallic filaments, different form of the specimens had to be chosen. Standard specimens would be not only hard to produce with appropriate precision but the main problem lies in the two finalizing steps – debinding and sintering. During these steps, parts with walls thicker than 4 mm are susceptible to occurrence of cracks and large deformations. Moreover, support structures must be manufactured from the source material. In our case, no separation material is available as it was descried for Markforged and Desktop Metal solutions. In the end, the 1BA shape from ISO 527 (Plastics — Determination of tensile properties) was employed (Figure 3).

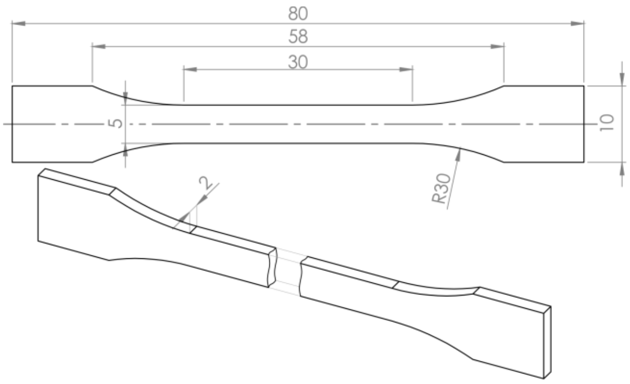

Figure 3. Dimensions of tensile test specimen

In the case of tensile testing, seven groups of samples were created. Overview of individual groups together with brief description is indicated in Table 2.

Group designation	Description	Number of samples
Laser	316L rolled material Laser cut from sheet plate	5
Water	316L rolled material Water cut from sheet plate	5
SLM_AB	SLM-printed 316L specimens As-built condition	5
SLM_M	SLM-printed 316L specimens Finished by milling	5
UTF_BASF	Ultrafuse 316LX Fabrication of specimens: TUL Debinding and sintering: BASF	5
UTF_TUL	Ultrafuse 316LX Fabrication of specimens: TUL Debinding and sintering: TUL	5
UTF_Vibrom	Ultrafuse 316LX Fabrication of specimens: TUL Debinding and sintering: Vibrom	5

Table 2. Overview of tested groups

First two groups marked *Laser* and *Water* refer to samples from 2 mm thick 316L stainless steel sheet plate. The shape given in Figure 3 was cut from this sheet by laser beam and water jet, respectively. Thanks to these two groups, it is possible to compare mechanical properties of BASF Ultrafuse 316LX products with standard rolled material. Use of water jet can also eliminate influence of heat on the value of standard mechanical constants which may be present for laser cutting.

Following two groups marked *SLM_AB* and *SLM_M* denote specimens fabricated using Selective Laser Melting technology and 316L stainless steel powder (supplier SLM Solutions Group AG, Lübeck, Germany). For both of these groups, the specimens were built in horizontal position using SLM280HL machine. For *SLM_AB* group, the only operation after finishing of the job was removal of support structures and thus this state represent the as-built condition. In the case of *SLM_M* group, given samples were finished using milling operations. Table 3 summarizes SLM process parameters which were used for fabrication of specimens from 316L stainless steel powder.

Parameter	Value
Laser power	175 W
Scanning speed	750 mm/s
Layer thickness	0.03 mm
Hatch distance	0.12 mm

Table 3. SLM process parameters for AISI 316L steel.

Last three groups refer to main topic of this article – the Ultrafuse 316LX filament. All the samples were fabricated at Technical University of Liberec using Felix Tec4 machine (FELIXprinters, IJsselstein, The Netherlands) with technological parameters listed in Table 4. Specimens were fabricated in horizontal orientation.

Parameter	Value
Nozzle size	0.5 mm
Extrusion multiplier	1.06
Extrusion width	0.4 mm
Retraction distance	1 mm
Retraction speed	45 mm/s
Layer height	0.15 mm
Outlines	2
Infill	100 %
Outline overlap	30 %
Nozzle temperature	245 °C
Bed temperature	80 °C
Default print speed	30 mm/s

Table 4. Technological parameters used for production of specimens from Ultrafuse 316LX filament.

In Figure 4, a snapshot from fabrication process of cubic sample from Ultrafuse 316LX material is shown. During the filament extrusion, liquefied material creates very dense compound which frequently sticks to the lower surface of extrusion nozzle. The material itself is very abrasive and thus it demands utilisation of a special hard nozzle or its replacement after a few extrusion cycles.

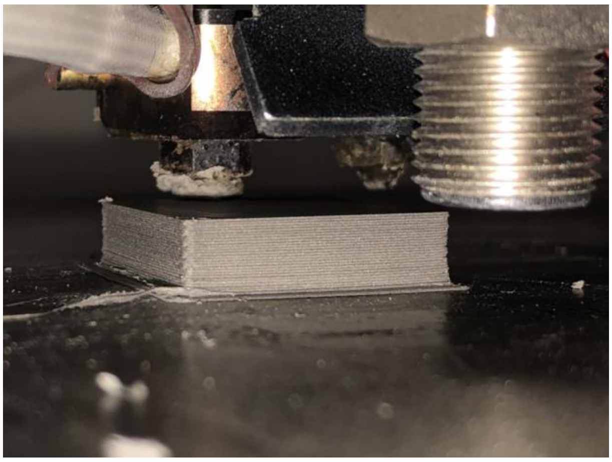

Figure 4. Extrusion of Ultrafuse 316LX filament on Felix Tec4 machine.

For the following steps, debinding and sintering, three different suppliers were tested. Firstly, manufacturer of the filament BASF offered to arrange finishing operations. This group is marked *UTF_BASF*. In the second case, finishing operations were done at the premises of Technical University of Liberec, hence the abbreviation *UTF_TUL*. Catalytic debinding was done in custom-made chamber using small doses of nitric acid. Overview of the whole device can be seen in Figure 5. The chamber itself is composed of 3.7 l glass jar and the inner plate made of 304L stainless steel which holds the products during the whole process. For better evaporation of the acid, the jar was heated to temperature of 80 °C. Side product of the debinding process - the inner gas was continuously removed from the jar into an exhaust system.

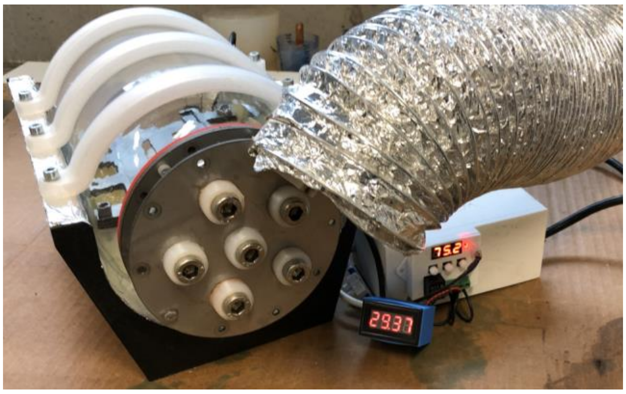

Figure 5. Custom-made chamber for debinding.

Consequently, products were submitted to sintering in vaccum furnace using 1380°C process temperature. Last group named *UTF_Vibrom* refers to finishing in the company Vibrom s.r.o. (Třebechovice pod Orebem, Czech Republic) which is a Czech leader in Metal Injection Moulding (MIM) technology. This technology uses similar post-processes described in this article. Moreover, the company frequently operate with source products from BASF. Thus, a possibility to use Vibrom's knowledge and equipment were tested for finishing of Ultrafuse 316LX products. In Figure 6, all the additively manufactured specimens are displayed.

Figure 6. Additively manufactured tensile test specimens. 1-SLM_AB, 2-SLM_M,3-UTF_BASF, 4-UTF_Vibrom, 5-UTF_TUL

1.2 Conditions of tensile tests

All the specimens underwent tensile test for determining the basic mechanical properties of tested materials. Initial tests failed due to slippage of the specimen from standard pneumatic clamping system. This was caused because of small clamping area of the specimen in combination with low value of clamping force. To solve this problem, special clamps were designed and manufactured from DIN 1.2709 tool steel using Selective Laser Melting technology. Using this process, it was possible to fit inner part of the clamp directly to the R30 radius (see Figure 3) between clamping and testing part of the specimen.

Tensile tests were carried out on TiraTest universal testing frame, equipped with ± 100 kN force transducer HBM (Hottinger Brüel & Kjaer GmbH, Germany). Each test was position-driven with defined speed rate of the machine's crosshead. Strain was measured with the use of MFL 800-B (MF Mess- & Feinwerktechnik GmbH, Germany) extensometer with initial distance L_0 = 15 mm between the blades. Before the test itself, preload of 300 N was applied to ensure proper position of the specimen in the clamps without any clearance. After this step, blades of the extensometer were attached to the specimen (Figure 7). In the initial part, constant speed rate of 1 mm/min was applied. After reaching 2 % strain, the speed was gradually increased up to 15 mm/min and this value remained constant until rupture of the specimen.

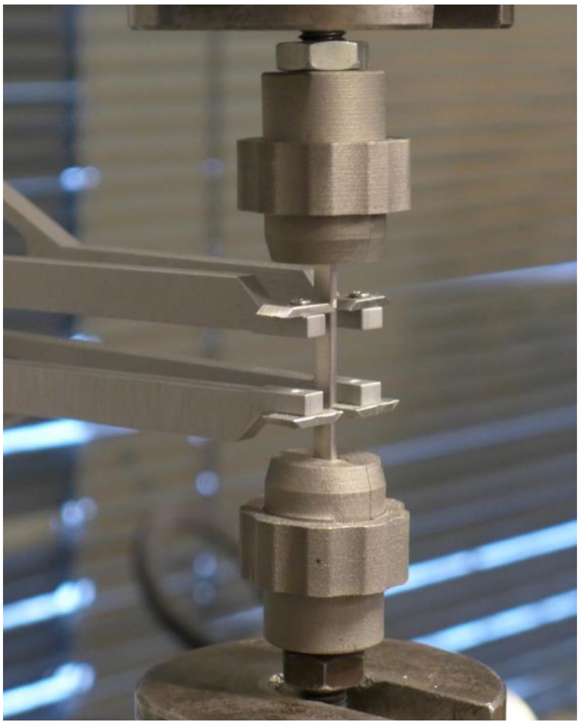

Figure 7. Tensile specimen in custom made clamps

RESULTS

For the first two groups of specimens which were extracted from 316L sheet plate (supplier Pronton s.r.o., Liberec, Czech Republic), the behaviour during the test is almost identical. Tensile curves for all the five tests from the *Laser* group are shown in Figure 8. As apparent, the curves almost overlap each other. The samples show no change not only for standard stress levels such as Ultimate tensile strength but also the value of strain at break. This behaviour was observed for both laser and water cut specimens.

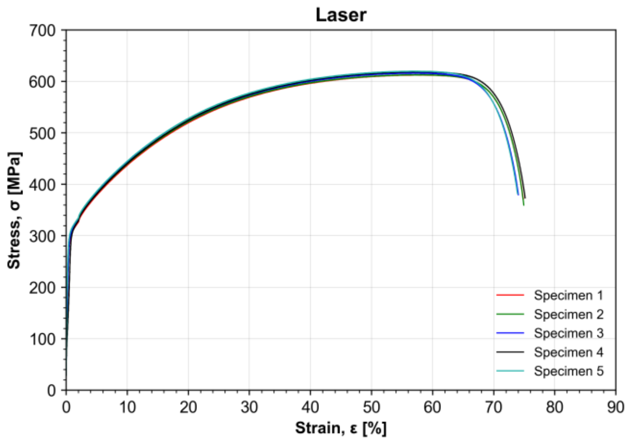

Figure 8. Tensile curves for samples from 316L sheet plate, cut by laser.

In the case of SLM-printed specimens, more scatter in the data can be seen. This is especially visible for strain at break value. As an example, stress-strain curves in Figure 9 are plotted for the specimens in as-built condition. In comparison with previous group, the ultimate tensile stress value is lowered. This may be caused by coarse surface of the unfinished specimens.

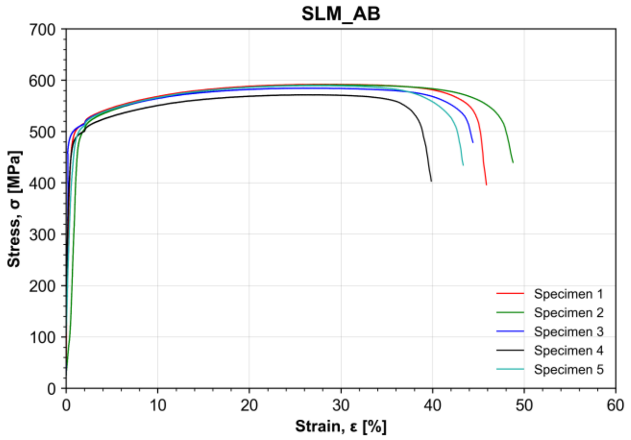

Figure 9. Tensile curves for samples fabricated with the use of SLM technology in an as-built condition.

BASF Ultrafuse 316LX specimens, which were post-processed in manufacturer's facility, show large strains before the rupture occurs. The value of strain at break reaches almost 90 %. Repeatability of the test is also outstanding as the only major difference can be seen at the end of stress-strain curves (Figure 10).

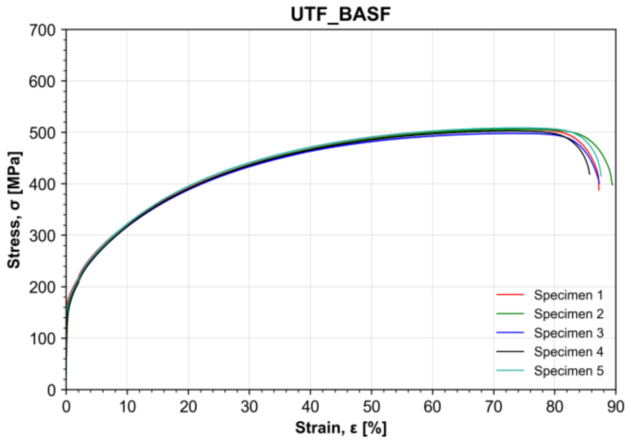

Figure 10. Tensile curves for Ultrafuse specimens, post-processed in the BASF company.

Due to the space limitations of this paper and better comparison of tested groups of specimens, standard mechanical constants were derived from related stress-strain curves. In Figure 11, Offset Yield stress $R_{p0,2}$ [MPa] and Ultimate Tensile Stress R_m [MPa] are displayed in the form of bar graph.

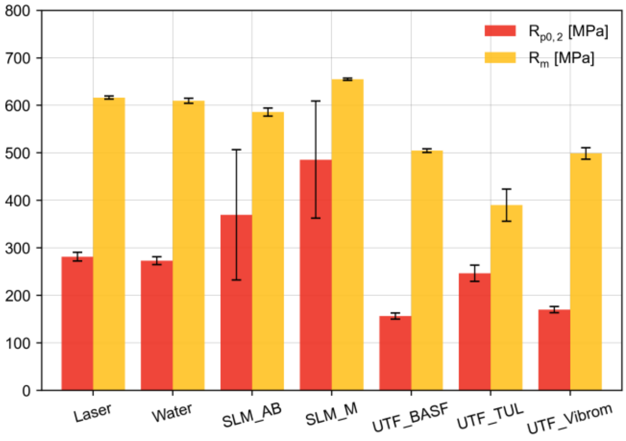

Figure 11. Ultimate Tensile Stress R_m and Offset Yield Stress $R_{p0,2}$. Mean value and standard deviation from five measurements.

Figure 12 show the same type of graph for strain at break ε_b [%] quantity.

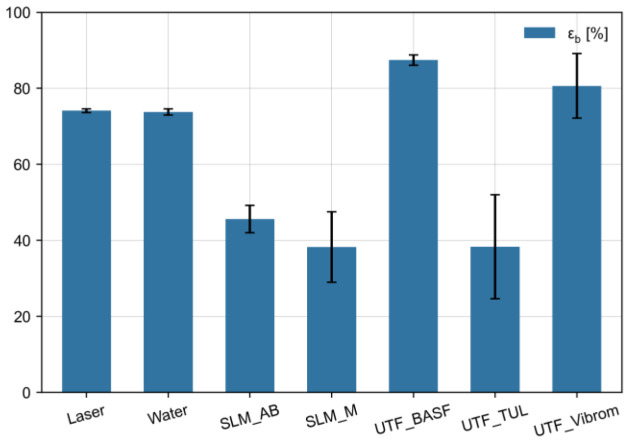

Figure 12. Strain at break ε_b. Mean value and standard deviation from five measurements.

DISCUSSION

In Ultrafuse 316LX material list from BASF Company, it is possible to find basic mechanical properties of the material [Forward AM 2020]. Similarly, SLM Solution Group AG presents standard material constants for 316L stainless steel in their material sheet [SLM 2020]. According to these materials, the tensile tests were done in agreement with DIN EN ISO 6892 for Ultrafuse material. SLM Solutions Group does not state any standard under which the tests were done. Mechanical properties found in above mentioned materials are summarized in Table 5.

Material	Tensile strength R_m [MPa]	Yield strength $R_{p0.2}$ [MPa]	Ductility A [%]
Ultrafuse 316 LX	561	251	53
SLM 316L	620	505	43

Table 5. Material constants for tested materials derived from manufacturer datasheet.

When comparing results of mechanical tests, several outcomes can be stated. First of all, specimens from rolled 316L steel show almost identical behaviour regardless of the method by which they were cut from source sheet plate. Results are consistent in both strength and ductility values.

Ultrafuse 316LX does not reach the strength of here tested rolled material. Standard stress values are more than 100 MPa lower than for the specimens from sheet plate. The values of Yield stress and Ultimate tensile stress does not correspond with data from the manufacturer. Again, the values are about 60 MPa lower for here tested specimens. Interesting finding is that with lower value of strength, the finished Ultrafuse 316LX product offers almost 40% higher value of ductility in comparison with values from datasheet. In this view, the 3D printed samples overcome even the rolled material. Two groups of specimens which underwent debinding and sintering in BASF and Vibrom Company show very similar performance. On the other hand, the specimens finished at TUL evince considerably worse mechanical behaviour. This is most probably caused by lack of dedicated equipment and non-typical process of debinding. Final structure may contain traces of binder and thus the final performance is limited. This topic needs deeper analysis for finding where the problem is and adjustment of finishing methods.

In the case of SLM-printed specimens, the results follow data published by the manufacturer. The material itself is more brittle than both rolled material and Ultrafuse 316LX. This phenomenon might be influenced by subsequent heat treatment, specifically solution annealing [Waqar 2020] [Kamariah 2020].

CONCLUSION

Main aim of this article was a direct comparison of FFF-manufactured Ultrafuse 316LX material with rolled material of the same kind and SLM-processed specimens. Secondly, the possibility of using the FFF technology for production of fully functional metallic parts was evaluated. In the frame of this work, it was decided to fabricate tensile test samples using commercially available BASF Ultrafuse 316LX material. Apart from this material, standard 316L steel was also tested for a possibility to compare final behaviour of FFF-produced samples with the conventional ones. Last set of specimens was produced using Selective Laser Melting technology for which the 316L powder is one of today's standard materials. Even this group serves as an interesting model for comparison of the data with another additive technology for manufacturing of metal parts.

One of the main finding of this paper is that it is possible to use very cheap desktop printers for fabrication of metallic models, more specifically the semi-finished products. BASF Ultrafuse 316LX Filament was easy to process after fine tuning of FFF technological parameters provided by the manufacturer. As a result, initial investment for fabrication of metal parts can be affordable for large number of companies. For the following two finishing steps, one has to be equipped with specific devices for debinding and sintering. In this view, the price of final model rises significantly because of need of relatively expensive and demanding machines. On the other hand, this can be also solved by submitting green part to a company which ensures the whole post-processing. This way was also applied in this work as two sets of specimens were submitted to BASF and Vibrom companies.

Tensile tests revealed that the material Ultrafuse 316LX does not reach values of ultimate tensile stress and yield stress typical for rolled 316L material. On the other hand, it offers more ductility which may be advantage in certain applications. Overall, these kinds of materials offer promising possibilities for further research and development of new types of design. Following work will thus be carried out to test other properties such as shrinkage, shape precision, porosity and deeper material-related analyses.

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