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Experimental evaluation of mechanical strength parameters of additively manufactured food-grade AISI 316 stainless steel

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Abstract

Direct metal laser sintering (DMLS) has become one of the most widely used methods for the additive manufacturing of metallic machine parts and tools. It allows the usage of all available metals and metallic alloys, enabling the production of parts with significant geometric complexity, which other technologies cannot produce. Many engineers, however, express concerns regarding the mechanical strength and reliability of items produced via DMLS. This article presents the results from an experimental evaluation of the basic strength parameters of samples of AISI 316 stainless steel, which were additively produced by means of selective laser sintering. Possible redistribution of the key alloying elements and changes in the phase state, which could result from the laser's thermal action and compromise the steel's corrosion resistance, have also been investigated using SEM and EDS. The experimental results revealed that the 3D printed parts show lower tensile strength by 13 % and lower modulus of elasticity by 57 %. The residual elongation after breaking and cross-contraction is also lower. The additively manufactured parts were also revealed to be porous, which could be seen as a disadvantage in the food processing industry.

1. Introduction

The rapid progress of computing and informational technologies throughout the last two decades has led to significant improvements in the working conditions for engineers and designers across the globe. The modern digital tools, in conjunction with some impressive hardware achievements, have paved the way for the implementation of a great variety of innovations within the scope of the manufacturing process for machines, different equipment, machine parts and tools. Throughout the years of progress in mechanical engineering, designers have always been limited by the capabilities of the available technologies for production.

The expansion and the optimisation of modern computing hardware, along with the impressive accomplishments in CAD software, have paved the way for the rapid advancement of the concept of additive manufacturing, which was initially conceived in the middle of the 20th century [1]. This has resulted in the introduction of several principles for the production of single machine parts, as well as units assembled by multiple parts, by adding material and building actual physical objects, replicating the geometric configuration and characteristics of virtual three-dimensional models. Because the procedure resembles the process of designing a document and printing it on paper, the technologies for additive manufacturing have become widely popular under the term "3D printing" [2,3].

The rapid growth and advancement of technologies for additive manufacturing have made them applicable to all types of materials used in mechanical engineering [2-5]. Three-dimensional printing allows the creation of machine parts that stand out with considerable geometric complexity, which other conventional technologies cannot achieve, or resorting to other methods related to

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severe obstacles that would come with considerable expenses. The technologies for additive production have lifted nearly all the limits for present-day engineers, granting them freedom of creativity with almost no boundaries [1-6].

Direct metal laser sintering (DMLS) is currently the most popular and widely used method for the additive manufacturing of metallic machine parts or assembled units [3-9]. Unlike other methods based on injection moulding, some of which use the same principle as fused material deposition (FDM), selective laser sintering does not require additional equipment for further heat treatment and/or chemical processing [5,7,8].

The source material comes as a very fine powder, which meets all the specific requirements needed for sintering. A special device spreads a thin deposit of this powder and tamps it to compact and uniformly distribute it with an equal thickness. A laser beam then performs the so-called "scanning" within the specific geometric constraints of each layer of the part being built. As the laser heats the metallic particles, partial melting occurs, forming microscopic welds. When a layer is complete, it is covered with another deposit of the powder, which is spread and tamped. The laser then scans this second layer and fuses the particles. This process is repeated layer after layer until the part is finished [6,8-10].

When this method is introduced to mechanical engineers as an available option, some questions arise. The designers usually have common concerns regarding the strength of machine parts produced this way. These concerns stand on the fact that this technology is based on the principle of sintering, and sintered parts generally show weaker resistance to external mechanical loadings. They are also porous, containing a substantial amount of microscopic air pockets, which is associated with some fragile behaviour [10-12].

The opportunities that additive technologies provide greatly benefit the food processing industry [13,14]. When it comes to producing parts (spare parts, moulds, tools, auxiliary equipment, etc.) that are meant to operate in direct contact with food products, rigorous hygienic requirements apply, some of which are incorporated in the international laws, regulations and standards [14-16]. The materials which come in contact with food products shall not contain any components in their chemical composition, which could migrate into the food products and become a prerequisite for health hazards. They must withstand aggressive chemical substances and sustain their properties at high temperatures – sometimes in conjunction with high pressure [14-16]. Their surfaces shall be smooth without pores. Rough surfaces, which feature open-air pockets, are difficult for sanitation as they retain remnants of food, which could undergo spoilage and become a favourable environment for developing harmful bacteria [15-17].

Austenitic stainless steels are one of the most common and desired materials for structural elements and auxiliary tools for equipment for manufacturing and processing food products and drinks. The literature currently includes many articles published by researchers who have studied the mechanical properties of additively manufactured metallic parts. Most of them, however, focus on instrumental steels such as 17-4 PH. The mechanical properties of additively produced samples of AISI 316 have been investigated. However, the methods used for their production were based on the extrusion of two-phase suspension, which consists of fine metal powder suspended in a thermoplastic binder. After the part is built, the binder is removed chemically or by burning before the sintering, which is done in separate equipment with a protective atmosphere [18,19].

A paper by Kim [20] describes a study carried out on 3D printed samples of AISI 316L. However, this study aimed to examine the effects of neutron and ionising radiation to which the samples were subjected before testing. Moreover, there has been no reported investigation on the compatibility of DMLS 3D printed pieces of AISI 316 with the hygienic requirements associated with direct contact with food products that are meant to be consumed by humans and/or their household pets.

This paper presents the results of an experimental study, which was carried out on 3D printed samples made of food-grade austenitic, non-magnetic stainless steel AISI 316. The primary goal of this study was to evaluate the mechanical performance of these samples and compare it with the performance of samples made of the same material using commercial machining technology. A secondary goal was further to inspect the structure of the additively manufactured samples, checking for the presence of open pores, which could severely compromise the hygienic performance of the steel. A possible heat-induced diffusion and redistribution of the key alloying elements caused by the thermal action of the laser was also investigated using energy-dispersive spectroscopy (EDS) analysis along with scanning electron microscopy (SEM).

2. Material and methods

Two groups of cylindrical samples intended for static uniaxial tensile testing at room temperature were made for the current study. Each batch included three test pieces. The first batch of samples was produced using commercial machining technology (CNC turning), while those in the second batch were made using DMLS additive technology. The samples, which were 3D printed, did not feature the centre holes shown in Figure 1. Additionally, the M10 threads on them were made using a commercial tool (thread-cutting die).

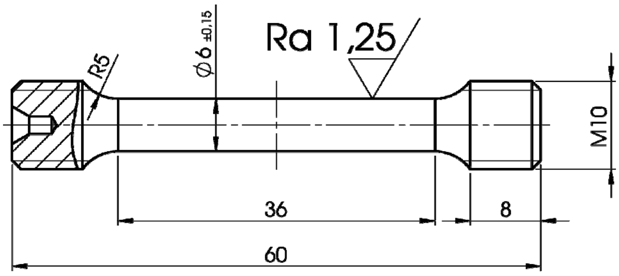

Figure 1. Test piece used for conducting uniaxial tensile test

The samples, which were not additively manufactured, were produced from a commercially available rod, whose initial diameter was 11 mm. The machining parameters such as cutting speed, cutting feed and depth of cut were all chosen on the grounds of desired final roughness of *Ra* = 1.25 µm and ± 0,15 mm tolerance, considering the requirements in EN ISO 6892, as the tensile testing was done in accordance with the cited standard. The additively manufactured samples were built from a spherical powder provided by 3D Systems Inc. (USA). The size of the particles was stated to be no greater than 50 µm. The wavelength of the laser is 1070 nm, operating at 300 W power. The layer thickness was 40 µm.

All the samples were of austenitic grade, non-magnetic stainless steel AISI 316 (EN 1.4401/X5CrNiMo17-12-2). Its typical chemical composition includes (wt. %): 16 – 18 Cr, 10 – 14 Ni, 2 – 3 Mo, approximately 2 Mn and nearly 0.75 Si. Small amounts of P, S and N are also present.

The tensile testing was carried out on modified Alfred J. Amsler & Co. equipment. The modification comes down to installing a tensometric sensor and a raster sensor. The loading rate was 0.75 mm/min, which is in accordance with ISO 6892. The measured temperature was 22 °C.

Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) were used to study the surface morphology generally produced by the realisation of this additive technology, as well as the potential redistribution of the alloying elements as a result of the thermal action of the laser beam. For this purpose, a single sample was made of the same stainless steel. It was a flat plate with dimensions 10 × 10 × 2 mm. No mechanical treatment (like polishing) was done on the sample, simply because the goal of the study required its surface features to be preserved as much as possible. In order to remove possible contamination, before placing the sample in the vacuum chamber of the scanning electron microscope, it was cleaned with acetone using an ultrasonic bath, which operated at a frequency of 20 kHz.

SEM was also used to study the fracture morphology from a randomly selected additively manufactured sample subjected to tensile testing. No treatment was done on this sample before placing it in the vacuum chamber of the SEM equipment. The SEM images were all obtained in a high vacuum, using 20 kV accelerating voltage and 110 µA current.

3. Results and discussion

The average values for the basic mechanical properties determined from the uniaxial tensile tests are given in Table 1. The values in brackets are the corresponding standard deviations. The stress-strain diagrams obtained from the conducted experiments are given in Figure 2.

Table 1. Average values of the mechanical properties for samples produced by DMLS and by CNC turning

Parameter	Produced by DMLS	Produced by CNC turning	Difference, %
Modulus of elasticity, GPa	12.5 (*SD* = 1.42)	29.3 (*SD* = 4.31)	57.34
Tensile strength, MPa	572.3 (*SD* = 37.08)	660.6 (*SD* = 55.69)	13.34
Elongation at break, %	32.8 (*SD* = 2.32)	41.1 (*SD* = 2.36)	20.19
Yield strength, MPa	467.6 (*SD* = 70.56)	518.5 (*SD* = 55.89)	9.82
Contraction at break, %	71.9 (*SD* = 4.25)	80.7 (*SD* = 5.56)	10.90

SD – standard deviation

The results show that the test samples produced by additive DMLS technology generally stand out with somewhat lower values for all the assessed mechanical properties. They tend to sustain much more elongation before breaking, as the nature of the breaking itself is more consistent with that of a fragile material. It occurs with the formation of a faint, barely visible neck around the place of breaking. On the other hand, the samples made by commercial machining on CNC equipment do not elongate that much. Once the maximum registered value for the stress is reached, a well-defined neck starts to form around the location where the breaking shall occur. This is a typical behaviour for a material with distinctive plasticity.

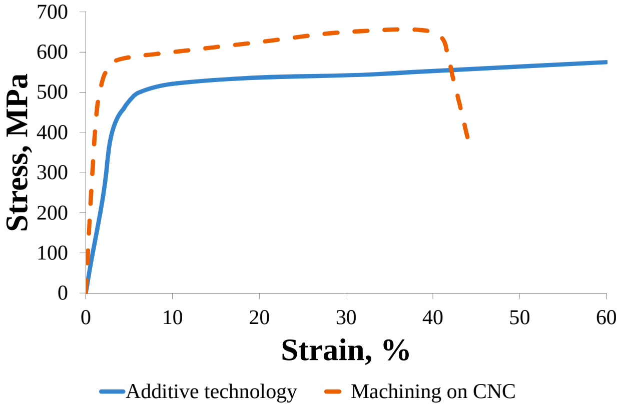

Figure 2. Stress-strain curves of DMLS samples and samples produced by commercial machining

The microstructure of the steel, which the samples are made of, consists typically of one phase only. This is the austenitic phase, which has a face-centred cubic crystal frame. It is usually characterised by improved and quite distinguishable plasticity characteristics in contrast with steels having single-phase ferrite or dual-phase ferrite-pearlite structures [11,12,21,22]. It can be seen clearly from the curves in Figure 2 that the rate of hardening is almost identical for the samples from both batches. The 3D printed test samples show lower values for residual plastic elongation (elongation at break). Despite that, before breaking, while they are still subjected to the axial loading force, the degree of relative deformation is higher, which is consistent with the smaller modulus of elasticity. This indicates that the samples made by applying additive technology are more elastic in behaviour than those made by commercial machining on CNC equipment. Nevertheless, the results clearly show that selective laser sintering produces parts that can sustain plastic deformation. Thus, the plasticity of the source steel is largely preserved.

Figures 3 and 4 show SEM images of the fracture of a tested sample produced by additive DMLS technology. These images are obtained by the secondary electrons detector (SED). Multiple pores are clearly seen on the fracture, confirming that the internal structure of the 3D printed parts is nonhomogeneous and filled with numerous air pockets. The SEM image on the surface of the 3D printed flat sample, which was acquired with the backscattered electrons detector (BSED), is given in Figure 5. It clearly shows a single-phase structure and rather uniform distribution of the components of the alloy. A specific characteristic of this class of non-magnetic Cr-Ni stainless steels is that when heated above a specific temperature (roughly about 500 °C), the chromium starts to migrate towards the surface of the austenitic grain [11,12,21-23]. This occurs due to two main reasons.

The first reason comes down to the high tendency of the chromium to form carbides (mainly $Cr_{23}C_6$) on the boundaries of the austenitic grains. Because of the different diffusion rates for the chromium and the carbon, the crystallites end up depleted with respect to Cr near their surfaces. Thus, the corrosion

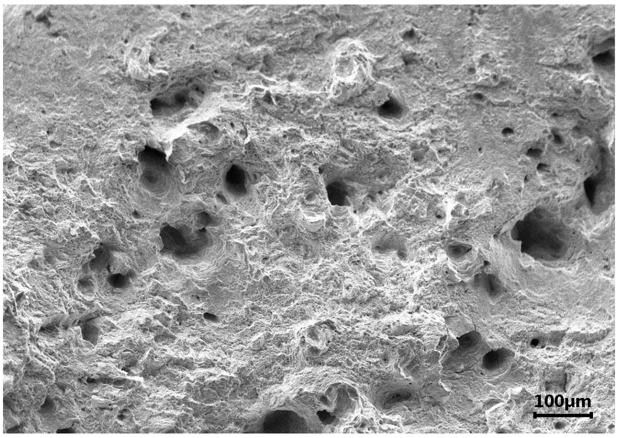

Figure 3. SEM image of a fracture on a 3D printed sample at 150×, SED

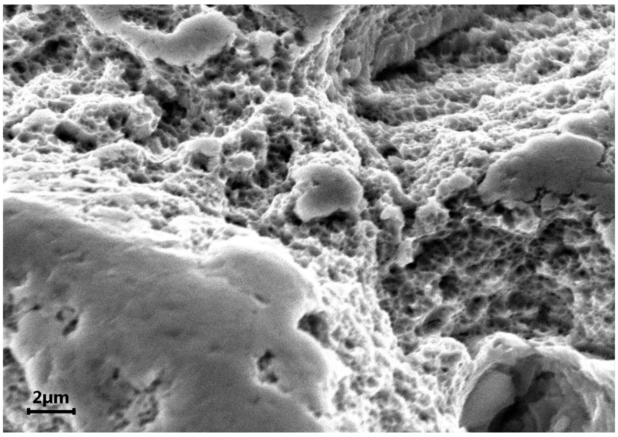

Figure 4. SEM image of a fracture on a 3D printed sample at 6000×, SED

resistance is compromised, and the distribution of the alloying components within the grain becomes non-uniform. The second reason is related to the affinity of the chromium to interact with the oxygen in the air. Because of this, it migrates to the surface of the steel, forming a protective transparent layer of Cr_3O_2, which is also known as the "passive layer" [21,23,24]. Since the laser heats the powder particles almost instantly and the cooling takes place quickly enough, the conditions for these changes are apparently absent. Moreover, the production of the part happens inside the working chamber of the equipment, where inert atmospheric conditions are maintained. A constant circulation of the gases inside further assists the cooling process. Numerous formations with predominantly spherical shapes are scattered on the surface (Figs. 5, 6 and 7). Such spheroidal objects are usually a result of condensation of metallic vapours. The laser beam heats up the metal and causes partial melting and some vaporization. This gaseous phase, which is formed, condenses into small spheroidal droplets, which end on the surface where they undergo a further phase transition into a solid state [25-27].

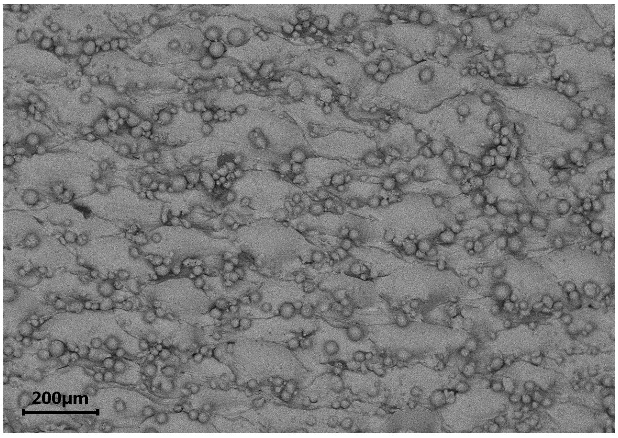

Figure 5. SEM image of the surface of a 3D printed sample at 100×, BSED

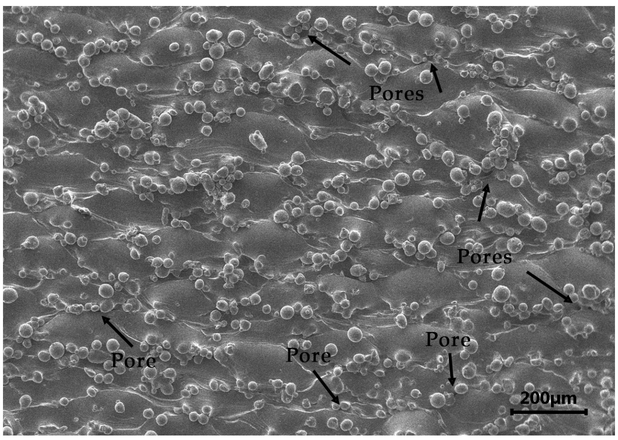

Figure 6. SEM image of the surface of a 3D printed sample at 100×, SED

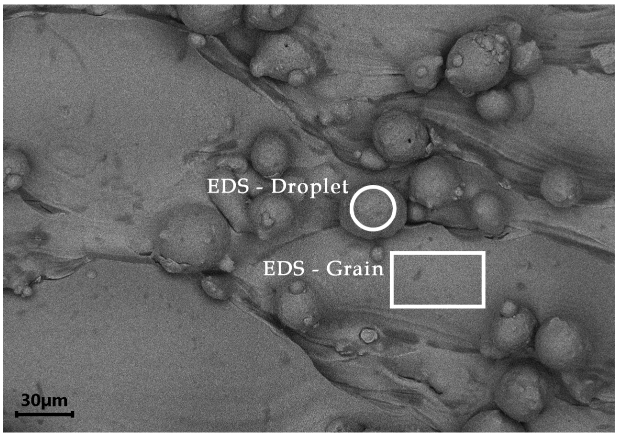

Figure 7. SEM image of numerous spherical formations seen on the surface of the 3D printed sample at 500×, BSED; EDS analysis locations are annotated separately

Some dendritic structures are also seen (Fig. 8), which result from the crystallization of the formed liquid phase under the peculiar thermodynamic conditions created by the action of the laser beam. The image obtained with the BSED shows a relatively uniform distribution of the alloying elements.

EDS analysis was performed in order to check the chemical composition of a grain (powder particle) and compare it with that of a spherical formation from a condensed and fallen droplet of metal. The purpose was to check if the vaporization, the subsequent condensation and solidification were accompanied by processes (such as oxidation and selective vaporization) which could result in the alternation of the initial composition of the alloy. The locations where the EDS analysis was performed are shown in Figure 7. The results of the EDS analysis regarding the elements of significance are given in Table 2, while the obtained spectrums are shown in Figures 9 and 10. No substantial changes in the chemical composition of the alloy have occurred.

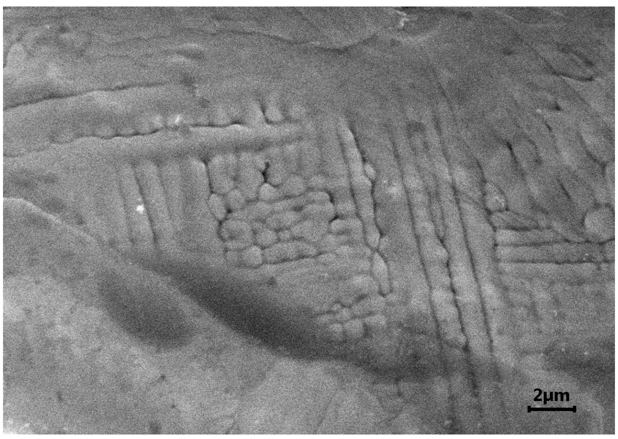

Figure 8. SEM image of dendritic structures seen on the surface of the 3D printed sample at 6000×, BSED

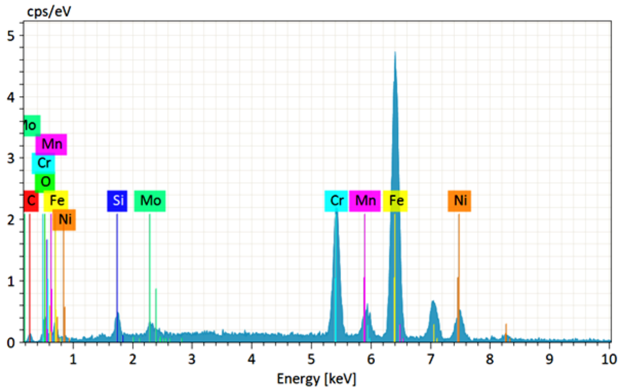

Figure 9. Spectrum obtained from EDS analysis of the grain (powder particle); location is denoted in Figure 7

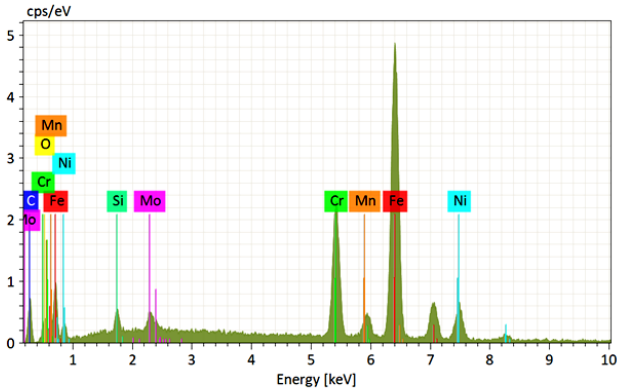

Figure 10. Spectrum obtained from EDS analysis of a spherical droplet; location is denoted in Figure 7

Table 2. Results of the EDS analysis on a single grain and a spherical droplet

Element	Concentration, wt. %				
	Cr	Ni	Mo	Mn	Si
Grain	17.05	9.03	2.71	2.32	2.19
Droplet	15.35	9.31	2.26	1.12	0.98

Pores could also be seen in the structure (some are pointed in Figure 6). Few randomly chosen air pockets are additionally examined under greater magnification (Figs. 11 and 12).

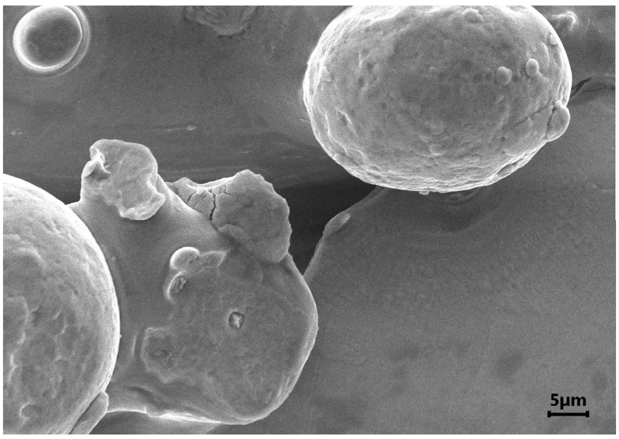

Figure 11. SEM image of a pore on the surface of the 3D printed sample at 2000×, SED

Figure 12. SEM image of a pore on the surface of the 3D printed sample at 2000×, SED

The SEM images of the fracture of the tested sample (Figs. 3 and 4) and the images from the surface of the flat sample (Figs. 5, 6, 7, 11 and 12) clearly reveal a porous structure of the 3D printed samples. Such structure, as well as the lower tensile strength and modulus of elasticity, are consistent with the nonhomogeneous composition of machine parts, which are produced using powder metallurgy [28,29].

4. Conclusion

An experimental evaluation was carried out for the basic mechanical properties of samples from non-magnetic austenitic stainless steel produced using direct metal laser sintering additive technology. The values for these characteristics were compared to the corresponding values for samples made of the same steel by using commercial machining technology.

The results show that additive technology can produce parts with sufficient mechanical strength regarding static loading at room temperature. Satisfactory plastic behaviour was also observed, which suggests the possibility of further processing such parts by means of plastic deformation.

The chemical composition of the additively produced samples was consistent with that of the base material. No redistribution of the key alloying elements, which would compromise the corrosion resistance of the steel, was seen to occur as a result of the thermal effects triggered by the action of the laser beam. The technology, however, appears to create porous and nonhomogeneous parts, which, in specific circumstances, could cause fragility of the manufactured items.

The DMLS technology's ability to produce parts that meet the rigorous hygienic requirements regarding parts intended to be used in direct contact with food products was also assessed. The observed open pores on the surface could trap food remnants, which could undergo spoilage and develop undesired bacteria. Sanitation also becomes difficult when a surface features exposed air pockets. Numerous approaches to counteract this setback of DMLS 3D printing could be implemented, and their efficiency could be additionally investigated. These include hot isostatic pressing, cold isostatic pressing, immersing the parts in molten metal, filling the pores with resins and applying surface coatings, which could seal the pores. The materials used to fill the air pockets shall also meet the same hygienic requirements regarding their chemical composition, resistance to aggressive substances and ability to withstand high temperatures.

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