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Study of Materials Produced by Powder Metallurgy Using Classical and Modern Additive Laser Technology

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Abstract

The paper focuses on an analysis of materials produced by modern classic additive method direct metal laser sintering (i.e. powder metallurgy). Direct metal laser sintering belongs to file of additive technologies of rapid prototyping. Rapid prototyping technology is a progressive group of methods used for fast creating of models, prototypes and components directly from 3D data. According to a used technology photopolymers, thermoplastics, specially modified paper or metal powders are used in rapid prototyping machines. Nowadays those production machines creates not only models and prototypes, but they are used to manufacture tools, forms and components for small series production.

The purpose of the theoretical part is to gain in depth understanding of exactly what is the principle of direct metal laser sintering and what are properties of explored materials. The practical part analyses structures of materials used by classic and powder metallurgy. It concentrates on samples preparation, their analysis (using light and electron microscopy) and testing. Samples are analysed by mechanical tensile test and Vickers hardness test. The paper also compares sintered samples with steel equivalents produced by classic metallurgy giving final recommendation.

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Keywords: Rapid Prototyping; Direct Metal Laser Sintering; Manufacturing Machine; Metallographic Analysis; Mechanical Properties

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1. Introduction

DMLS is a revolutionary RP additive method. Which is becoming more a more important and nowadays it is considered to be the most perspective method for fast instrument applications. The method has still been developing. Currently this manufacturing machine is able to directly sinter metal powders (i.e. powders which are not covered with a plastic or other material with low melting point). Layers of standard metal powders with a low melting point contain phosphorus that is harmful to most of metals even in small quantity. Therefore there exist relatively hard construction limits. Material qualities are from now on a limiting factor to spread this method. A range and qualities of available materials have still been improving [1, 2, 3, 4, 5, 6, 7].

A principle of additive method DMLS Fig. 1 is based on the gradual melting of ultra-line layers of metal powder using a laser beam. A production of prototype component starts by pre-processing when a machinist load STL data of a model to software of manufacturing machine EOSINT M 270. Software designed for preparation of data for manufacturing machine EOSINT M 270 is very easy. It enables easily and quickly control validity of input data and with help of clear graphics interface it is possible to define location in working space, support type etc. The next step is a choice of building powder material and appropriate thickness of manufacturing layers considering precision/resolution and manufacturing speed. The service software assigns right technology parameters of building and "cut" 3D data into component layers. Data are subsequently sent to the manufacturing machine EOSINT M 270. A steel platform is fixed to a working chamber of manufacturing machine. The component is built on the steel platform a batching plant sets needed powder amount for one layer and a lift arm with a ceramic edge spreads a uniform layers on surface of s steel platform according to the set thickness. Control software of manufacturing machine controls variable focusing of a laser beam and a beam trajectory along component outline. The metal powder is totally melted in the place of the laser beam fall where a bottom layer is "melted throw-out" and subsequently it solidifies [2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13].

The paper goal is to verify if powder materials (corrosion resistant steel EOS GP1, martensitic steel EOS MS1) processed by additive method DMLS are their mechanical properties fully comparable to materials produced by classic conventional technologies. Testing samples are produced by manufacturing machine EOSINT M 270 within an analysis. They are chip machined, analysed (light and electron microscopy), tested (mechanical tensile test and Vickers hardness test) and compared with steel equivalents - X5CrNiCuNb16-4 (1.4542), X3NiCoMoTi18-9-5 (1.2709) produced by classical metallurgy.

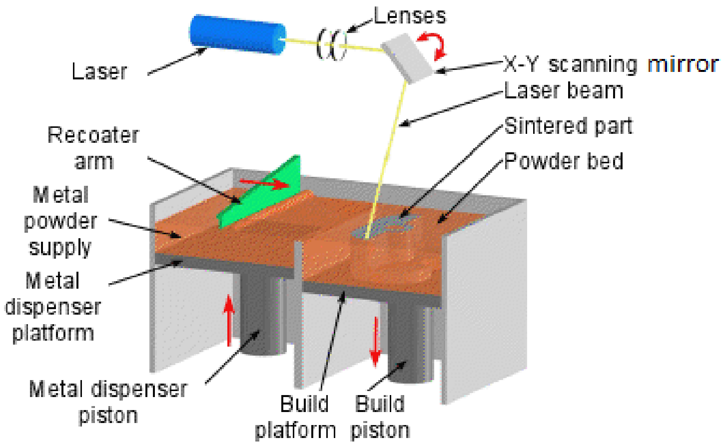

Fig. 1. Principle of DMLS Method [3, 14].

2. Experiment

The practical part presents material analyses and shows results of several mechanical tests. Analysed materials are GP1 EOS, EOS MS1 for EOSINT systems and materials X5CrNiCuNb16-4 (1.4542) X3NiCoMoTi18-9-5 (1.2709) produced by conventional metallurgy.

2.1. Description of Analysed Materials

Stainless Steel GP1 is alloyed stainless steel in the form of a fine powder. The chemical composition corresponds with the U.S. classification 17-4 PH and European classification 1.4542. This steel type is characterized by very good corrosion resistance and good mechanical properties especially excellent ductility. Chemical composition of steel is described in Table 1.

Table 1. Chemical Composition of Steel [Weight %] [15, 16].

C	Si	Mn	Mo	Ni, Cu	Cr	Nb
Max. 0.07	Max. 1.00	Max. 1.00	Max. 0.50	3.00 – 5.00	15.00 – 17.50	0.15 – 0.45

Corrosion-resistant steel X5CrNiCuNb16-4 is a martensitic precipitation hardening steel stabilized by niobium (Nb). The excellent corrosion resistant of steel is comparable to corrosion resistance of austenitic steel. When the steel is heated it is resistant to inter granular corrosion and erosion in corrosive environment as well. Chemical composition of steel is in Table 2.

Table 2. Chemical Composition of Steel [Weight %] [17].

C	Si	Mn	P	S	Mo	Ni, Cu	Cr	Nb
Max. 0.07	Max. 0.70	Max. 1.50	Max. 0.040	Max. 0.015	Max. 0.60	3.00 – 5.00	15.00 – 17.00	5 times C up to 0.45

MS1 Tool steel appears in the form of a steel powder. Its composition corresponds with the European classification 1.2709 and German classification X3NiCoMoTi 18-9-512. It has excellent mechanical properties. Heat treatment is very easy. When steel is simply heat-hardened (hardening at 490°C for 6 hours) it receives high hardness and stability. Chemical composition of steel is in Table 3.

Table 3. Chemical composition of Steel [Weight %] [15, 16].

C	Si, Mn	P, S	Cr	Mo	Co	Ni	Ti
Max. 0.03	Max. 0.15	Max. 0.010	Max. 0.50	4.50 – 5.20	8.50 – 9.50	17.00 – 19.00	0.60 – 0.80

Tool steel X3NiCoMoTi18-9-5 is high-strength martensitic stainless steel suitable for work under extreme cold and hot temperatures. Therefore tool steel is appropriate to highly stressed components for aeronautics industry and rocket technologies, pressure vessels, gears, moulds for plastic manufacturing and tools for pressure casting. Chemical composition of steel is in Table 4.

Table 4. Chemical composition of the steel [Weight %] [17].

C	Si, P, S	Cr	Mo	Co	Ni	Ti
Max. 0.03	Max. 0.10	Max. 0.25	4.50 – 5.20	8.50 – 10.00	17.00 – 19.00	0.80 – 1.20

3. Tests of Mechanical Properties

Tensile test and Vickers hardness test were carried out to indicate mechanical properties.

3.1. Tensile Test

Uniaxial tensile test of materials belongs to basic tests. It is pre-determined to become the most widespread and accepted testing method for evaluating the mechanical properties of materials. All because of its principle of simplicity and functionality [18, 19]. Tensile test was carried out according to IEC 10002, ISO 6892-1 and DIN 50 128.

The comparison of EOS GP1 material produced by DMLS method with material X5CrNiCuNb16-4 (1.4542) produced by conventional metallurgy is shown in Table 5 and Fig. 2. Curves of tensile diagrams show that the material GP1 exhibits especially higher tensile strength about 400 MPa. Yield strength is almost 400 MPa lower. Contraction is 45% lower, elongation values are identical.

Table 5. Measured Values – Material 1.4542 and GP1.

Material	S_0 [mm^2]	$R_{p0.2}$ [MPa]	F_{max} [N]	R_m [MPa]	A [%]	Z [%]
GP1	28.18	564.29	41,906.96	1,489.10	11.69	15.67
1.4542	28.18	1,019.86	29,659.79	1,068.05	10.91	62.55

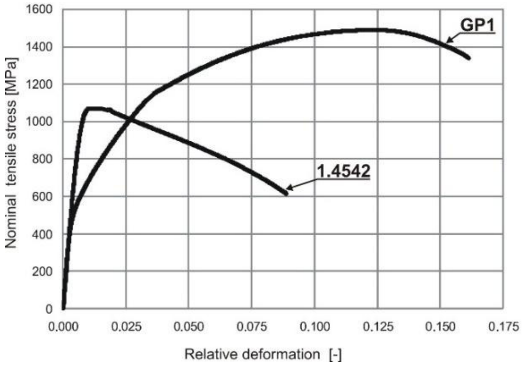

Fig. 2. Nominal Tensile Stresses of Tested Materials (1.4542, GP1).

The comparison of material MS1 produced by DMLS method with material 1.2709 produced by conventional metallurgy is shown in Table 6 and Fig. 3. The tensile diagram curves show material MS1 exhibiting higher values of examined parameters than material 1.2709 (the tensile strength is about 200 MPa higher and the yield strength is about 70 MPa higher). Contractions are 25% higher, elongation values are almost identical.

Table 6. Measured Values – Material 1.2709 and MS1.

Material	S_0 [mm^2]	$R_{p0.2}$ [MPa]	F_{max} [N]	R_m [MPa]	A [%]	Z [%]
MS1	28.18	901.94	35,903.61	1,274.51	14.28	49.37
1.2709	28.18	833.32	29,634.91	1,050.84	15.35	74.42

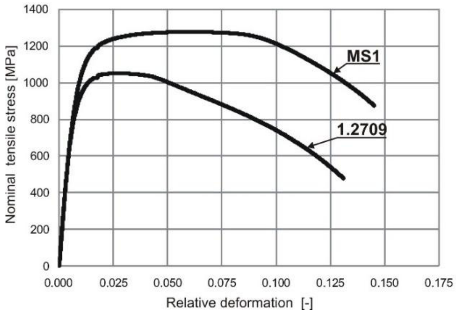

Fig. 3. Nominal Tensile Stresses of Tested Materials (1.2709, MS1).

4. Vickers Hardness Test

Hardness measurement was processed according to the standard ISO 6507 [18, 19]. It was carried out under room temperature (20 ± 5°C). The load testing was done for 10 HV.

The hardness comparison of materials GP1 and MS1 made by DMLS with materials produced in a standard way is shown in Fig. 4 and Fig. 5.

The graph comparing materials GP1 and 1.4542 shows visible increase of hardness in deformed (strengthened) sample part of GP1 material in comparison with the sample of material 1.4542, whereas hardness values are almost constant throughout the whole sample length. To determine actual sample hardness it is important to focus on the undistorted sample part where hardness values of GP1 material is about 60 HV higher.

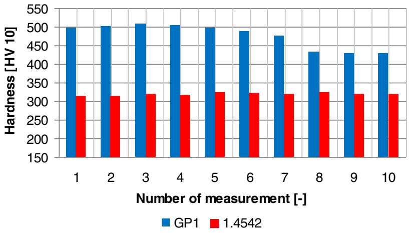

Fig. 4. Comparison of Hardness (HV 10) Values of Materials GP1 and 1.4542.

The comparison of materials MS1 with 1.2709 indicates that hardness values are almost constant throughout the sample of both materials. Yet to determine actual hardness, values are compared in the undistorted head of test sample whereas MS1 material hardness is about 60 HV higher.

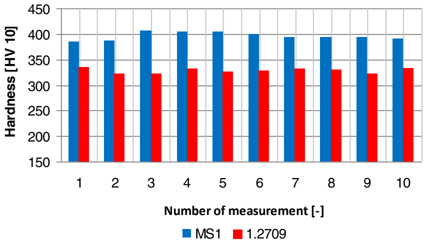

Fig. 5. Comparison of Hardness (HV 10) Values of Materials MS1 and 1.2709.

5. Metallographic Analyses

For exploration and recording of materials structures the light microscope Olympus GX 51 with digital camera and the scanning electron microscope Philips X L30 are used. The metallographic analysis compares structures of explored materials. It compares materials micrographs from light and electron microscopy. Fig. 6 a), b) shows a ferritic-pearlitic microstructure where ferrite partially shows Widmannstatten morphology. The structure in Fig. 7 a), b) is made of martensite and pearlite [2, 20, 21].

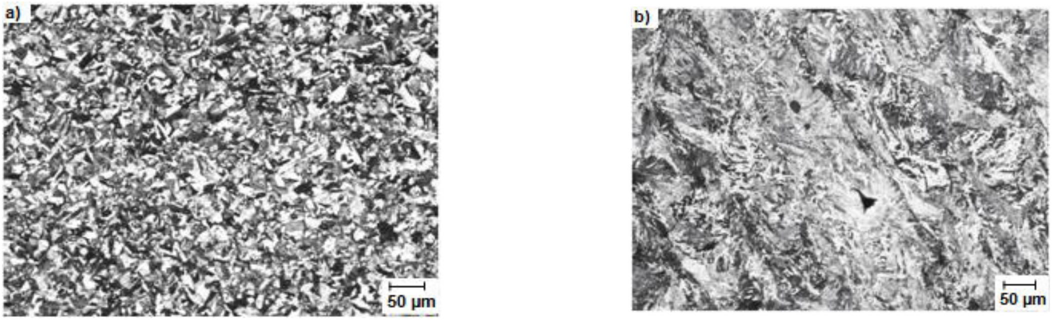

Fig. 6 (a) Micrograph of Material 1.2709 (Marble); (b) Micrograph of Material EOS MS1 (Marble).

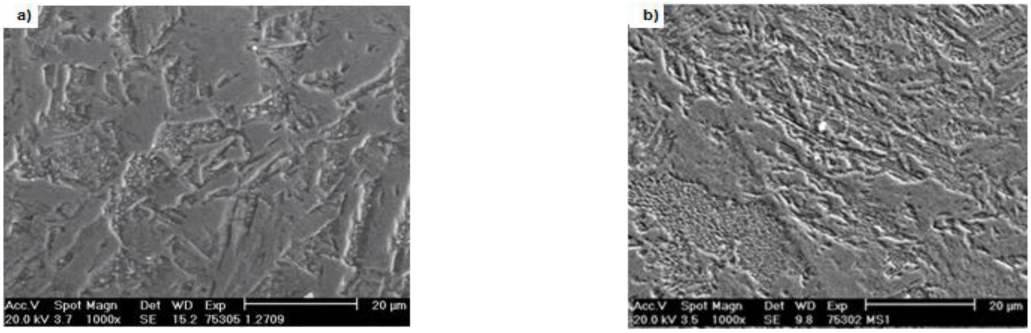

Fig. 7. (a) Micrograph of Material 1.2709 (Marble); (b) Micrograph of Material EOS MS1 (Marble).

Fig. 8 a), b) and Fig. 9 a), b) show the martensitic structure and residual austenite.

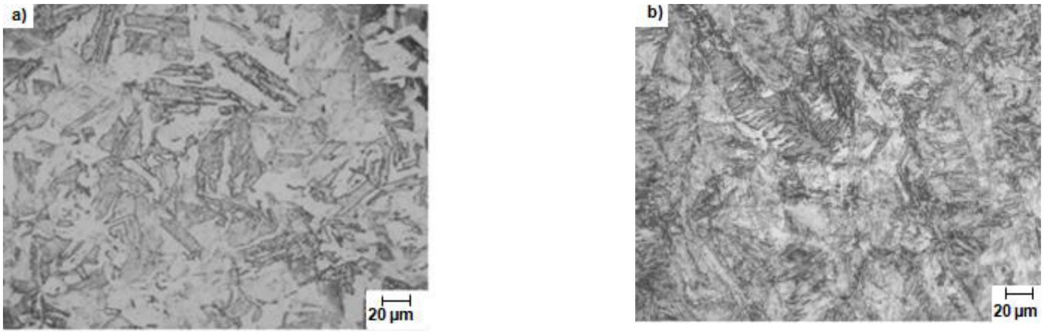

Fig. 8. (a) Micrograph of Material 1.4542 (Marble); (b) Micrograph of Material EOS GP1 (Marble).

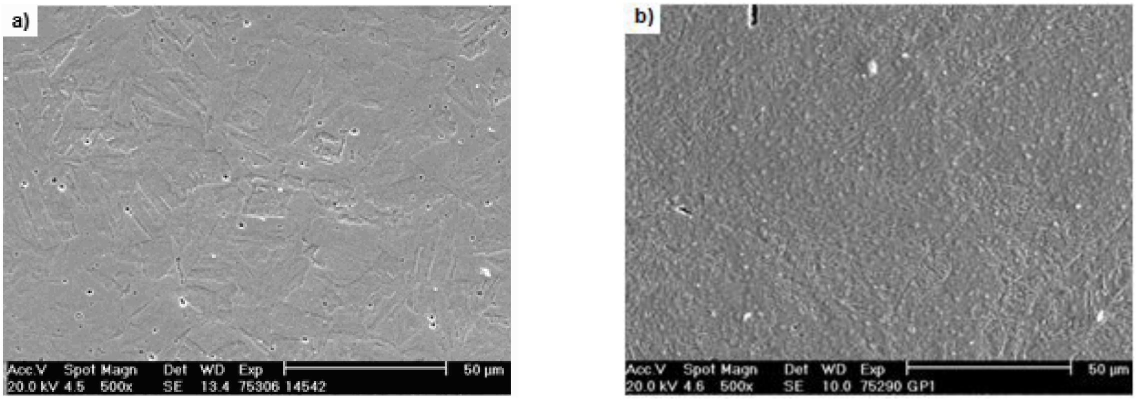

Fig. 9. (a) Micrograph of Material 1.4542 (Marble); (b) Micrograph of Material EOS GP1 (Marble).

6. Image Analysis

An image analysis is done in images gained by light microscopy. Porosity is explored on EOS GP1 and EOS MS1 materials. An analysis of porosity is carried out by PMG 3 light microscope with DP 20 digital camera using Stream Motion software. Fields of view are photographed to analyze porosity see Fig. 10 a) and Fig. 10 b). Final values for individual fields are shown in Table 7.

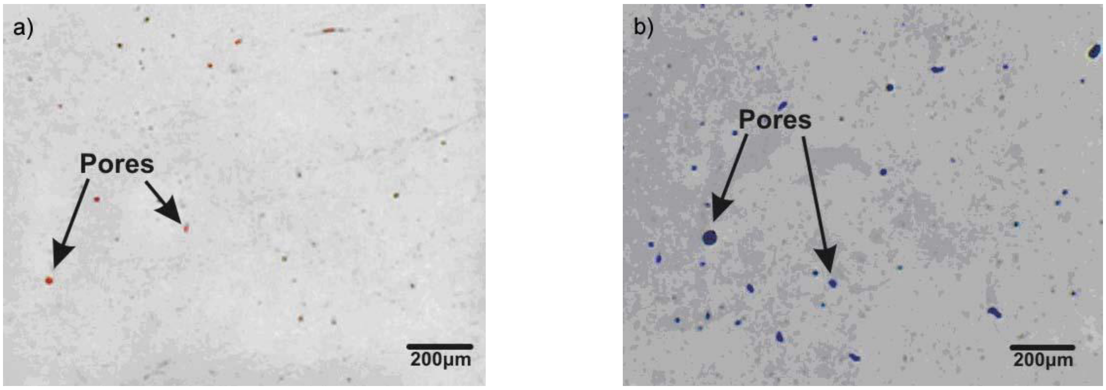

Fig. 10. (a) Analyzed area – Material EOS GP1; (b) Analyzed area – Material EOS MS1.

Table 7. Final Parameters and Porosity of Analyzed Materials – GP1, MS1.

Material	Cut	Shape Factor [-]	Vertical Elongation [-]	Horizontal Elongation [-]	Ratio of max. to min. dimension [-]
GP1	Longitudinal	0.81	0.36	2.24	1.91
	Perpendicular	0.79	0.37	2.05	1.81
MS1	Longitudinal	0.68	0.38	2.16	1.86
	Perpendicular	0.90	0.45	1.87	1.66
Material	Cut	Maximum Diameter	Average Dimension	Area	Porosity

		[μm]	[μm]	[μm^2]	[%]
GP1	Longitudinal	12.06	7.74	3,863.52	0.05
	Perpendicular	10.41	6.75	3,972.41	0.05
MS1	Longitudinal	15.69	10.37	16,824.39	0.45
	Perpendicular	12.78	8.60	22,573.05	0.38

The pores shape has significant influence on stress concentration and stress risers. Pores in the structure mainly have sphere shape. Therefore there are fewer stress concentrators and stress risers in the total volume. Porosity does not show any direct effect caused by deposition and melting of individual layers. Porosity appears at very low levels in the same values for both vertical and horizontal direction. It can be assumed that even this low porosity should have a negative effect on mechanical properties as R_m, $R_{p0.2}$, A. However tensile tests of materials do not confirm this fact. The comparison with materials 1.4542 and 1.2709 produced by standard technology (i.e. rolling) shows higher values of R_m, $R_{p0.2.}$ Elongation almost corresponds only yield strength of GP1 material is lower. In conclusion, porosity of materials GP1 and MS1 made by DMLS does not have a major impact on mechanical properties [22].

7. Discussion on Experimental Results

Final individual parameters from mechanical tests of materials (corrosion-resistant steel 1.4542 and tool steel 1.2709 produced by standard metallurgy and materials EOS GP1 and EOS MS1 made by DMLS) indicate that mechanical properties correspond to material sheets. It is important to note that materials are not after heat treatment. References indicate materials 1.4542 and 1.2709 as chemical equivalents of sintered materials EOS GP1 and EOS MS1 [2, 14, 15].

The comparison of tensile test results with Vickers hardness results shows that mechanical values of materials produced by DMLS are higher than values of the 1.4542 and 1.2709 materials. It indicates that materials produced by conventional metallurgy are only the chemical equivalent with different mechanical properties. For example yield strength of EOS GP1 material is about 400 MPa higher and slip limit is about 400 MPa lower compared to 1.4542 material.

The metallographic analysis shows a similar micrograph of analysed samples. Materials produced by DMLS show an increased number of cracks and discontinuities of complex impurities in comparison with conventional materials.

Porosity of materials EOS GP1 and EOS MS1 is analysed as well. Porosity of the materials produced by standard technologies cannot be monitored because they are rolled materials in which porosity does not occur. Porosity values of EOS GP1 material vary in hundredths of percent of area cut and for EOS MS1 material in tenths of percent of area cut.

Conclusion

Analysis and comparison of particular material structures of classic and powder metallurgy shows following partial conclusions:

- Results of tensile test indicate that parameters (R_m, $R_{p0,2}$, A, Z) of materials produced by additive method DMLS are not the same as parameters of materials produced by classic methods. Some parameters are higher, e.g. R_m value of material EOS GP1 is 400 MPa higher and oppositely $R_{p0,2}$ value is 400 MPa lower.
- Vickers hardness test shows that hardness values of materials produced by additive method DMLS are 60 HV higher than values of classic materials. Measured average hardness of EOS GP1 is 428 HV and for EOS MS1 is 394 HV.
- The same phases of fine-grained martensite and residual austenite are located in structures of material EOS GP1 produced by both classic and additive method DMLS. A microstructure of material EOS MS1 is formed by martensite and perlite where material 1.2709 is formed by ferrite-pearlite microstructure.

- When additive method DMLS is applied porosity of material EOS GP1 varies in hundredths of percent and for material EOS MS1 varies in tenths of percent. They are mostly spherical pores bringing no crimped effects too material volume. That claim confirm above mentioned performed mechanical tests.
- Performed analyses indicate that testing samples produced by additive method DMLS do not contain almost no pores and inclusions but there exist some exceptions.
- Samples produced by additive method DMLS show huge amount of disruptions and complex dirt comparing to samples produced by classic metallurgy.
- Breaking morphology for both materials is the same. It assigns quasi-fissile character with transcrystalic break corresponding to analysed microstructures.

Performed analyses confirm that mechanical properties of powder materials EOS GP1 and EOS MS1 processed by additive method DMLS are fully comparable with materials produced by classic metallurgy. Carried tests prove that a production of prototypes or other components is fully replaceable by modern additive method DMLS.

DMLS method provides a wide application range of various qualities, from controlled porosity for bleeding or filtration up to fully homogenous structures that are able to reach higher solidity than casts and forged pieces.

Table 8 shows summary evaluation and comparison of specific parameters of tested materials.

Table 8. Mechanical Properties of Material Structures in Classic and Powder Metallurgy.

Material	Rp0,2 [MPa]	Rm [MPa]	A [%]	Z [%]	HV 10 [%]	Porosity [%]
MS1	901.94	1,274.51	14.28	49.37	394	0.41
1.2709	833.32	1,050.84	15.35	74.42	328	0
GP1	564.29	1,489.10	11.69	15.67	428	0.05
1.4542	1,019.86	1,068.05	10.91	62.55	320	0

In conclusion additive method DMLS is worth to be further and detailed analysed. The object of further analysis would be comparison of given materials after heat processing where interesting microstructures and mechanical properties are anticipated considering a development of new materials. The use of new or improved plastic and metal materials (e.g. with higher resistance, elasticity or solidity) that would lead to spreading this production method is anticipated in future. Gained results mentioned in the paper may in future help spreading additive method DMLS into awareness of wide public not only in Czech Republic.

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