Additive manufacturing (AM) has enabled not only designers over the last few years to boost a huge boom in the design and optimization of machine components. Part of the additive manufacturing is not only the design of the component itself, but the individual steps involved in the creation of computer documentation necessary for the preparation of the AM, the selection of suitable material and the printing itself with possible subsequent heat treatment. Process parameters, including the choice of powder, have a significant effect on the resulting component properties. The powder is one of the process features that play a significant role. The main aim of the research is to compare the properties of materials made from different powders manufacturers. A micro - tensile tests (M-TT), reinforced by metallographic and fractographic analysis, were used to analyse the materials, allowing a detailed discussion of the resulting material properties.

KEYWORDS
Additive manufacturing (AM), powder size and distribution, mechanical testing, maraging steel, porosity.

1 INTRODUCTION

The additive manufacturing process was found to be a breakthrough technology, not only for the design phase or prototype development. The process considerably reduces the manufacturing time needed to produce components from months to weeks [Wimpenny 2017]. The morphology and size of powders are important factors for powder bed fusion processes because they affect powder flowability, laser energy absorption and the thermal conductivity of the powder bed. Spherical particle morphology improves the flowability of the powder to achieve a high packing density in the powder bed, which improves the final quality of the SLM-processed parts. On the other hand, the flow of powder with a non-spherical shape is obstructed because the particles tend to interlock mechanically and entangle with each other. Hence non-homogeneous layers of powder with varying packing densities form on the top of the previously built solid surface, which may lead to the formation of defects such as porosity and/or incomplete melting. A reduction in particle size results in an increase in the surface area, which favours the absorption of laser energy to increase the melt pool temperature; an increase in the gap in the powder bed, which may lead to high porosity in the consolidated part if the gap is too large; an increase in the tendency for particle agglomeration; and a reduction in powder flowability [Brandt 2017].

As in many powder-based production processes, the flow and corresponding layer packing of the powder dictates the efficiency in selective melting and the quality of the product. After the manufacturing process, the non-molten powder is recycled and can be used for future printing [Murr 2012]. The apparent density of the powder, which influences the final density of the SLM parts, depends on the powder size, shape, and size distribution. Generally, packing of spheres leads to a higher density than other shapes. The spherical particles with smooth surfaces may also improve the powder flowability and deposition. Moreover, finer powders may result in a higher apparent density (to some extent), indicating a higher final density and mechanical properties. The SLM process is set by adjusting various parameters. An optimal combination of laser power, scanning speed, powder layer thickness, and scan line spacing (also known as hatch spacing) is required to minimize the potential defects (by achieving optimal melt pools) and to produce high quality parts [Srivatsan 2016].

2 MATERIALS AND METHODS

The comparisons of two maraging steel powder suppliers were the main objective of this research. The components designed for mechanical testing and metallographic analysis were produced under the same conditions. The built parts (Fig. 1) have a chemical composition corresponding to classification of maraging steel 1.2709, a material having very good mechanical properties, excellent hardness and strength. A total of 3 components were analysed and tested in as-built states. Two samples (sample no. 1 and 2) were produced from supplier 1 (S1) and one sample (sample no. 3) from supplier 2 (S2). The powders were new for samples 1 and 3, sample 2 was produced from powder with recycled fraction.

Figure 1. The shape of AM component designed in COMTES FHT for mechanical testing and metallographic analysis

2.1 Powder analysis

Powder particle size distribution is an important factor influencing the deposition and the SLM part density. The following figures show that the powder particles were analysed in terms of shape, dimensions and distribution. SEM images were analysed manually by means of image analysis in NIS Elements 3.2 digital image processing and analysis software. Powder particle size distribution is evident from the Tab. 1 and Tab. 2, summarizing the percentage of average sizes. The S1 powder contains a larger variety of particle sizes, occurrence of not only spherical particles, which exceeded their average values (Fig. 2, Fig. 3). was observed. The S2 powder particles (Fig. 4, Fig. 5) observed in this study contained higher proportion of smaller particles, with smooth surface and
regular, spherical shape. The highest share of the particle size was observed in a range of 15 - 25µm. A total of 500 particles from both manufacturers were measured.

Table 1. Powder particle size distribution – S1

| Class | Number | %Number | Cumulative | %Cumulatively |
|-------|--------|---------|------------|---------------|
| 0-5   | 8      | 1,6     | 8          | 1,6           |
| 5-10  | 52     | 10,4    | 60         | 12,0          |
| 10-15 | 84     | 16,8    | 144        | 28,8          |
| 15-20 | 80     | 16      | 224        | 44,8          |
| 20-25 | 81     | 16,2    | 305        | 61,0          |
| 25-30 | 61     | 12,2    | 366        | 73,2          |
| 30-35 | 56     | 11,2    | 422        | 84,4          |
| 35-40 | 36     | 7,2     | 458        | 91,6          |
| 40-45 | 27     | 5,4     | 485        | 97,0          |
| 45-50 | 6      | 1,2     | 491        | 98,2          |
| 50-55 | 5      | 1       | 496        | 99,2          |
| 55-60 | 1      | 0,2     | 497        | 99,4          |
| 60-65 | 1      | 0,2     | 498        | 99,6          |
| 65-70 | 2      | 0,4     | 500        | 100           |

Table 2. Powder particle size distribution – S2

| Class | Number | %Number | Cumulative | %Cumulatively |
|-------|--------|---------|------------|---------------|
| 35-40 | 11     | 2,2     | 499        | 99,8          |
| 40-45 | 1      | 0,2     | 500        | 100           |

2.2 Porosity and hardness measurement

Porosity was analysed by means of the Image analysis and the material exhibited very low porosity level of all samples. Although the level was generally low, the level was doubled for samples 2 in comparison to sample 3. Conventional hardness measurement was provided by means of standard Vickers hardness measurement. Properties, such as porosity and HV10 of analysed maraging steel from different powder manufactures is summarized in Tab. 3.

Table 3. Porosity level and hardness values

| Sample | Porosity | HV10 |
|--------|----------|------|
| 1      | 0,03 %   | 352  |
| 2      | 0,04 %   | 368  |
| 3      | 0,02 %   | 363  |

2.3 Microstructural analysis

Samples intended for metallographic analysis by light microscopy were cut according to the individual directions in relation to the direction of printing. The analysed sections in the individual observed planes underwent standard metallographic preparation. Microstructure was revealed with Picral agent and observed by light microscopy. 3D microscopic images in Fig. 6 and Fig. 7 document the maraging steel 1.2709 microstructures. The Z axis represents the direction of the printing. XY plane is a cross-section perpendicular to the direction of printing. The XZ and YZ planes show typical lines indicating the melt pools morphology (the “melt pools”). This
type of microstructure, which is similar to the directed solidification of the microstructure, is the result of epitaxial and dendritic grain growth in a direction identical to the direction of the heat flow. There were no significant differences in microstructure among both suppliers.

Figure 6. Supplier 1 - Sample 1

Figure 7. Supplier 2 - Sample 3

2.4 Mechanical testing

Mechanical properties of the powder bed processed metallic parts are strongly dependent on the microstructure and the relative density of the parts [Brandt 2017]. AM products exhibit strong properties dependence on the process parameters, e.g. position within the depositing chamber, building orientation, building direction, component volume. Consequently, measurement of local properties, which is for most cases impossible with the use of standard sized specimens, is a critical issue. In many cases, it is not possible to determine properties with standard sized specimens as the component is smaller than the size of standard specimens. Mini-specimen methods were developed especially for residual service life assessment [Dzugan 2017], [Dzugan 2018]. Micro - tensile testing procedure (M-TT) was developed in COMTES FHT based on the demand to measure reliably the material characteristics by the tensile tests from the minimum amount of the experimental material. The geometry of the test bodies is given in Fig. 8. The basis of the test is established on the standard ISO 6892-1. However, as these standards do not consider the testing of miniature bodies, the procedure with the internal regulation has been developed and accredited. Micro - tensile testing has proved to be effective in earlier studies [Dzugan 2017], [Dzugan 2018] and [Rund 2015 and Podany 2014] and the values of mechanical properties are fully comparable with the results of standard tensile tests. The tests were performed on a special LabControl test facility with a capacity of 5kN, accurate measurement of the deformation is provided by the Digital Image Correlation system (DIC).

Tensile tests of the miniaturized test specimens in the 0°, 45° and 90° directions relative to the printing direction were performed. Summarized values of the tensile test stress-strain characteristics (yield stress YS, tensile strength UTS, uniform elongation EU, elongation EL and reduction of area RA) are given in Fig. 14. The scatter in the results is reported and is attributed to the defects in the AM-processed components. Stress concentration effect caused by the loading direction being perpendicular to the orientation of the planar defects (lack of fusion) demonstrated the influence the sample orientation to the direction of testing (Fig. 9 – Fig. 10). Printing direction by sample 3 did not have significant effect on M-TT results, Fig. 11.

Figure 8. M-TT specimen geometry

Figure 9. Stress – strain curves Sample 1

Figure 10. Stress – strain curves Sample 2
2.5 Fractography

The scanning electron microscopy images of the as-built M-TT fracture surfaces of both suppliers in the 90° testing direction (Fig. 12 and Fig. 13) demonstrate the impact of defects on resulting mechanical properties. The red arrow in Fig. 12 marked a location with the presence of lack of fusion, as the energy was insufficient to melt all the powder. Lower ductility and contraction in samples from supplier 1 was caused due to the higher number of internal discontinuities and pores in the microstructure. Sample 3 (S2) proved lower number of pores and both ductility and contraction were not significantly affected. For all samples, a transgranular ductile fracture with typical dimple morphology was observed.

3 CONCLUSIONS

The study provided a detailed investigation of the maraging steel from two powder suppliers. The microstructure and local properties measurement with the use of M-TT technique proved that although components reached similar values of YS and UTS, the main difference was observed in the ductility. The presence of internal defects (lack of fusion, pore sites) detected on the SLM fracture surfaces of the test bodies may be the cause of such low values of mechanical properties, mainly the ductility by supplier 1. The recycling of the powder did not adversely affect the values of YS and UTS. The printing direction did not prove significant effect on the M-TT results of the
material from supplier no. 2. The research showed that components can be effectively characterized with the use of miniaturized specimens, as M-TT technique provides testing more sensitive to the material defects in the microstructure of additively manufactured materials.

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