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SLM Processing of 14 Ni (200 Grade) Maraging Steel

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Abstract

Selective Laser Melting (SLM) offers new possibilities in part manufacturing and many of these are of big interest for the tool- and die industry. Besides structural optimisation, further SLM-processable materials are currently in R&D focus, as the combination of structural improvements - such as conformal cooling for tooling applications – the use of application-specific materials can further improve the overall part performance. 14 Ni (200 grade) maraging steel is a die steel that ideally fulfills the requirements of the aluminium die casting process. High thermal conductivity, a low thermal expansion coefficient and stability of mechanical properties at elevated temperatures lead to an amelioration of tool performance. The paper presents a general characterization of 14 Ni (200 grade) maraging steel powder and its SLM processability. A comparison of material properties of additively and conventionally processed 14 Ni (200 grade) maraging steel.

1 Motivation

Selective Laser Melting (SLM) offers new possibilities in part manufacturing due to the layerwise build-up process. These opportunities are of big interest for many different industrial sectors and their applications, covering aerospace industry, medical engineering, jewellery and arts as well as the traditional branch of tool- and die-making. Besides structural optimisation and complex design R&D in SLM currently strongly focuses on the development of further SLM-processable materials [1]. An extension of the list of SLMprocessable materials will further push this manufacturing technology. This will also enable the implementation of this technology in other industrial sectors, since many industries intend to keep on working with their approved standard materials and alloys, at least in the beginning.

Toolmaking is a very experience based and traditional industrial branch but of significant importance for the European industrial landscape. While additive tool manufacturing is already state of the art for plastic injection moulding – especially to achieve conformal cooling as shown by Radig [2] – other, metal-working industries currently begin to think about additive manufacturing in general and particularly about SLM.

This paper reports on the SLM-processing of 14 Ni (200 grade) maraging steel, commercially known as Marlok[®] C1650, that – according to its manufacturer [3] – ideally fulfills the requirements of the aluminium die casting

process: high thermal conductivity, low thermal expansion coefficient and stability of mechanical properties at elevated temperatures.

These first results are on the general SLMprocessability, the fabrication of dense parts and the comparison of material properties of conventionally and additively manufactured 14 Ni (200 grade) maraging steel.

2 Experimental Setup

2.1 14 Ni (200 Grade) Maraging Steel Powder

Chemical Composition

The 14 Ni (200 grade) maraging steel powder that has been processed was produced by gas atomization from bulk material and delivered by Metso Minerals, Inc., Metso Materials Technology, Finland. The chemical composition of 14 Ni (200 grade) maraging steel according to the manufacturer is shown in Table 1.

¹ inspire AG, St. Gallen, Switzerland

Table 1: Chemical composition of 14 Ni (200 grade) maraging steel

Element	wt.%	Element	wt.%
С	0.013	Si	0.057
Cr	0.038	Mn	0.049
Мо	4.37	S	0.0047
Ni	14.5	Р	0.016
Со	10.98	Al	0.056
Ti	0.24	Cu	0.018

Particle Size Distribution

A decisive requirement regarding SLM-processability of a powder of some material is its flowability, since it enables the powder delivery by the coating device. The flowability is particularly depending on the particle form and size distribution of the powder. Table 2 displays the data delivered by the powder manufacturer in volume-% and the data measured at inspire. The latter data covers two values per size range: volume-% and number-% which enables a more comprehensive understanding of the powder. The data of the manufacturer and inspire match quite well and represent a typical distribution of SLM-processable powder. The analysis of the powder flowability - conducted on Revolution Powder Analyzer, Mercury Scientific Inc., Newton CT - results in a mean avalanche angle of 38.8 \pm 3.5°, and a mean surface fractal of 2.11 \pm 0.58. These values lead - according to the flowability analysis method developed by Spierings et al. [4] - to a flowability of φ =0.91 which is an excellent value for SLM-processability.

Table 2: Particle size distribution of 14 Ni (200 grade) maraging steel

Size range	Data	Data	Data
	Manufacturer	Inspire	Inspire
Particle dia.	Volume - %	Volume - %	Number - %
< 20 µm	23.23	19.6	49.4
20-32 μm	51.14	50.5	40.7
32-45 μm	25.46	27.4	9.0
>45 µm	0.17	2.4	1.0

2.2 SLM Process

Even though SLM is a highly complex manufacturing process and influenced by various physical effects, there are only four key machine parameters:

- laser power
 P_{Laser}
- scan speed v_{scan}
- hatch distance h
- layer thickness t

These four parameters can be put together to the following equation that calculates the volume energy density e - i.e. the heat input per volume element – according to [5].

$$e = \frac{P_{Laser}}{v_{scan} * h * t} \tag{1}$$

The SLM-experiments were conducted on a Concept Laser M2 machine that is equipped with a fibre laser with maximum output of 194 W at the build platform, operated in cw-mode.

2.3 Precipitation Heat Treatment

The heat treatment has been performed according to the manufacturer's guidelines for precipitation heat treatment [3], which is identical to the heat treatment of other maraging steels [6, 7]:

- Heating up to 525°C with maximum heating rate of 150°C/h
- Holding at 525°C for 6 hours
- Cooling down in the furnace according to free air cooling

3 Results and Discussion

3.1 Material Properties

Part Density

After the particle size distribution has been evaluated, other experiments were conducted to check the SLMprocessability of 14 Ni (200 grade) maraging steel. The general SLM-processability was analysed by a parameter study to identify the process window for manufacturing of dense and flawless parts, which is a key goal of the SLM-processability and of essential importance for applications of SLM-processed 14 Ni (200 grade) maraging steel.

With regard to the part density the volume energy density is a decisive factor. Figure 1 shows the relative part density in relation to cast or wrought material as a function of the applied volume energy density during the part's SLM production. The analysis was done according to Archimedes' principle based on 14 Ni (200 grade) maraging steel raw density of 8.09 g/cm³ that is given in the manufacturer's datasheet [3]. The resulting graph for 14 Ni (200 grade) maraging steel density analysis is shown in Figure 1 and is a characteristic trend line for steel powders that are SLM-processed. Each point in the graph is representing a machine parameter set. It is clearly visible that a certain amount

of energy input is inevitable to achieve acceptable relative part densities, i.e. > 99.0%. If higher energy input per volume is applied the relative part density obtained by SLM rises to a range of 99.1 - 99.3%. Since the trend line is not linear there is a limit in achievable density. For economic reasons the parameter set for production will be chosen such that sufficient part density can be realised with lowest energy input. The most suitable production parameter – the so-called "standard parameter" – has a volume energy density of 78.2 J/mm³. The corresponding value is marked with a black arrow in Figure 1, the respective relative density is 99.2%.



Figure 1: Relative part density – 14 Ni (200 grade) maraging steel; density analysis according to Archimedes` principle

Since the Archimedes' principle is based on a raw density value given by the powder manufacturer, further experiments have been conducted to proof this data provided. An optical assessment of some test samples has been performed. These samples were cut in their xzplanes with z being the direction of SLM build-up. The optical evaluation has been conducted on a Keyence VHX 100 digital microscope with 200x magnification. From each sample, twelve images have been taken randomly from the respective cross sections, and have been analysed with regard to pores. In Figure 2 the arithmetic means of optically analysed parameter sets are added to the characteristic of relative part density as a function of volume energy density. The optically analysed relative part density is higher for all assessed parameter sets, whereat the differences range from 0.25% - 0.77%. Spierings et al. [8] showed that with regard to accuracy the Archimedes' principle has evident advantages compared to an optical evaluation, since the latter is based on the information of one cross section whereas Archimedes' principle enables an overall statement. The comparison of the optically analysed material density with the Archimedes density allows matching the effective bulk material density to a value of 8.047 g/cm³. Further experiments were conducted to check the raw density of bulk 14 Ni (200 grade) maraging steel material, provided by the powder manufacturer. Its density was determined by the Archimedes' principle which resulted in 8.017 g/cm³. Secondly, this sample was ground to a defined geometry, weighted and then its density was calculated to 8.040 g/cm³. Subsequently the values for relative part density displayed in Figure 1 – based on raw density of 8.09 g/cm³ – are minimum values since an evaluation based on a raw density below 8.09 g/cm³, as it has been determined based on bulk material, would result in higher relative density values.



Figure 2: Relative part density – 14 Ni (200 grade) maraging steel; density analysis according to Archimedes' principle in comparison to optical analysis

Despite these uncertainties and deviations regarding the raw density of 14 Ni (200 grade) maraging steel it can be clearly stated that 14 Ni (200 grade) maraging steel powder is well processable by SLM, since the previously mentioned experiments have successfully been conducted on another machine setup – Concept Laser machine, type M1 – as well.

Hardness

The hardness of SLM processed 14 Ni (200 grade) maraging steel has been determined by HV_{10} on the measuring instrument Brickers 220 from Gnehm Härteprüfer AG. The samples were manufactured with the standard parameter having a relative density of 99.2%. Hardness analysis is based on eight samples that have been measured five times and in two directions respectively, xy-plane that is parallel to the building plane and zx-plane beeing vertically oriented to the building plane. The value in xy-direction is 360.7 ± 6.0 HV₁₀, and the value in z-direction is 364.1 ± 6.5 HV₁₀, respectively. While these results show statistically significant differences based on α =5%, the precipitation heat treatment that substantially increases the hardness, leads to an elimination of these differences, since the values of 493.0 \pm 9.1 HV₁₀ for xy-direction and 491.7 \pm 7.2 HV_{10} for z-direction do not show this statistical significance anymore. This effect can be explained by

the precipitation that develops isotropic and acts as dislocation barrier in all directions.

Static Mechanical Properties

For the analysis of the mechanical properties of SLMprocessed 14 Ni (200 grade) maraging steel tensile test samples with different specifications have been manufactured. Figure 3 illustrates the variation between the bars: a sample was produced in an upright, vertical orientation while the other two samples were oriented horizontally during the SLM process. For the latter two samples the orientation of the scanning vectors in form of islands relative to the bar axis was divided into two groups. In the first horizontally scanned sample the vectors were parallel and perpendicular to the bar axis – referred to as $0^{\circ}/90^{\circ}$ – whereas the directions changed to $+/-45^{\circ}$ for the second sample – referred to as $+/-45^{\circ}$. Additionally each of these three samples has been analysed based on five tensile bars respectively in its asbuilt condition and after the precipitation heat treatment.



Figure 3: left: vertically and horizontally oriented tensile bars during SLM build-up; right: horizontally manufactured tensile bar with different orientation of scanning vectors relative to the bar axis



Figure 4: Tensile properties of SLM-processed 14 Ni (200 grade) maraging steel in as built and in heat treated (HT) condition; HT according to the data sheet [3]

Figure 4 shows the results of tensile testing that reveal three notable facts that have been assessed qualitatively so far:

1. The results of the three samples in the as built condition show merely little variance with respect to the orientation of the tensile bars during SLM build-up; the mean values vary by less than 1.3 % for $R_{p0.2}$ and 5.3 % for R_m compared to one another.

2. The results of the three samples in the heat treated condition show merely little variance with respect to the orientation of the tensile bars during SLM build-up; the mean values vary by less than 3.9 % for $R_{p0.2}$ and 3.2 % for R_m compared to one another.

3. Values of all heat treated samples exceed the mean values of bulk material given in the datasheet [3] by more than 6.7 % for $R_{p0.2}$ and 3.4 % for R_m .

Observations 1 and 2 are particularly interesting since it is an inherent characteristic of the SLM process to produce parts containing a specific anisotropy due to the columnar microstructure.

Regarding the resulting Young's modulus E that is shown in Figure 5 it is notable that the vertical sample comes up with the lowest value. However, its value in the heat treated condition is in the range of the values given in the datasheet [3].



Figure 5: Young's modulus E of SLM-processed 14 Ni (200 grade) maraging steel

Thermal Conductivity

As mentioned in the introduction a beneficial property of 14 Ni (200 grade) maraging steel for its application in tooling components is its high thermal conductivity.



Figure 6: Thermal conductivity of SLM-processed 14 Ni (200 grade) maraging steel

Figure 6 shows the results of thermal conductivity measurements performed by Fraunhofer IWU using the half time method on a Laser Flash Apparatus from Netzsch. The chart starts at temperatures of 50 °C since the ambiences led to large variations and instabilities in the measurements in the temperature zone below 50 °C. The arithmetic mean of SLM-processed 1.2709 tool steel [9] in hardened condition is additionally added to the chart for comparison reasons. It is also worth mentioning that the measured data for SLM-processed 14 Ni (200 grade) maraging steel considerably exceeds the values provided by the powder manufacturer in the datasheet [3] for bulk material. The reasons for the substantial increase in thermal conductivity of SLM-processed 14 Ni (200 grade) maraging steel due to the

precipitation heat treatment is subject of ongoing research.

4 Conclusion

This paper presents the general SLM-processability of 14 Ni (200 grade) maraging steel powder on two SLM machine different setups. For ongoing investigations only one of these two systems has been selected for the conduction of the experiments. For the chosen machine the ideal process parameter set ensuring part density on one hand and productivity on the other hand requires a volume energy density of 78.2 J/mm³ delivered to the powder bed. Further promising results of SLM-processed 14 Ni (200 grade) maraging steel in comparison to additively manufactured 1.2709 tool steel were achieved and are summarized below:

1. Hardness is in a similar range to 1.2709 tool steel after precipitation heat treatment and it is not depending on the build-up direction of the part.

2. Mechanical properties after heat treatment are superior to the values given in the datasheet [3].

3. The tensile properties show merely little anisotropy in comparison to other SLM-processed steels as shown by Guan et al. [10] and Tolosa et al. [11]. This very interesting result will further be analysed by the authors – particularly based on microstructural images – to get a wider knowledge of the SLM-processability of 14 Ni (200 grade) maraging steel as well as of the SLM process in general since this is a controversial result to current understanding of the process.

4. Thermal conductivity measurements show promising results for an increasing spread of 14 Ni (200 grade) maraging steel as additively manufactured tooling material that unites its beneficial properties with the advantages of the SLM process.

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Literature

- [1] T. Wohlers, "Wohlers Report 2014," pp. 51-55.
- [2] G. Radig, "Werkzeugtechnik sorgt für verzugsfreie komplexe Bauteile," VDI-Z special Werkzeug-/Formenbau November, 2013, pp. 24-26.
- [3] Metso Powdermet Materials Technology Solutions, "Marlok[®] - Longer die life – Better quality," Datasheet, 2005.

- [4] A.B. Spierings, M. Voegtlin, T. Bauer, K. Wegener, "Powder flowability characterization methodology for powder-bed-based metal additive manufacturing," Progress in Additive Manufacturing Journal, 2015.
- [5] B. Vandenbroucke and J.-P. Kruth, "Selective laser melting of biocompatible metals for rapid manufacturing of medical parts," Rapid Prototyping Journal, vol. 13, no. 4, 2007, pp. 196-203.
- [6] M. Cabeza, G. Castro, P. Merino, G. Pena and M. Roman, "A study of laser melt injection of TiN particles to repair maraging tool steels," Surf. Interface Anal. (2014), ECASIA special issue paper.
- [7] S. Gulizia, D. Jones, M.Z. Jahedi and P. Koltun, "Thermal Fatigue Behaviour of Die Materials for Diecasting Tooling", Feature, pp. DC21-DC24.
- [8] A.B. Spierings, M. Schneider, R. Eggenberger, "Comparison of density measurement techniques for additive manufactured metallic parts," Rapid Prototyping Journal, Vol. 17, No. 5, 2011, pp. 380-386.
- [9] S. Polster, M. Gebauer, Experimental Data, 2015.
- [10] K. Guan, Z. Wang, M. Gao, X. Li, X. Zeng, "Effects of processing parameters on tensile properties of selective laser melted 304 stainless steel," Materials and Design, Vol. 50, 2013, pp. 581-586.
- [11] I. Tolosa, F. Garciandia, F. Zubiri, F. Zapirain, A. Esnaola, "Study of mechanical properties of AISI 316 stainless steel processed by "selective laser melting", following different manufacturing strategies," International Journal of Advanced Manufacturing Technology, Vol. 51, 2010, pp. 639-647.