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Microstructure and properties of SLM high speed steel

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Abstract

Selective laser melting (SLM) is a commonly used laser powder bed technique where the final properties are influenced by many different powder related properties, such as particle size distribution, chemical composition and flowability. In applications where high hardness, wear resistance, strength and good heat properties are required, high speed steels (HSS) are widely used today. HSS has high carbon content and are therefore considered as unweldable. The rapidly growing implementation of AM technologies has led to a growing range of new applications and demands for new alloys and properties. The interest in being able to manufacture HSS by SLM without cracking is therefore increasing. In SLM, it is possible to preheat the base plate to a few hundred degrees Celsius which has been used for HSS and proved successful due to reduced thermal gradients. In this study, the properties of SLM produced high speed steel PEARL Micro®2012 with a carbon content of 0.61 wt.-% have been investigated and compared to those of a forged and rolled PM-HIP counterpart ASP®2012.

Keywords

AM, selective laser melting, metal powder, high speed steel, microstructure and hardness.

1. Introduction

Selective laser melting (SLM) is a commonly used laser powder bed technique (LPBT). A thin layer of metal powder with a thickness of typically 20-100 µm is distributed on a building platform using a recoater device. Based on CAD data, a laser selectively melts the powder as well as parts of the previous powder layer to achieve fusion between succeeding layers. It is important that the thin layer of powder is spread to a reproducible layer with dense packing, even thickness and without voids to favour the final properties of the produced parts. The powder therefore needs to have the right properties, such as flowability, particle size distribution and tap density [1-2].

SLM is used in industries such as medical engineering, aerospace and automotive where the most commonly used steels are austenitic stainless steels AISI 316L and AISI 304, maraging steel 1.2709 and precipitation hardenable stainless steels 17-4PH and 15-5PH [3]. As the need to use additive manufacturing (AM) for new applications increases, new properties and thus new alloys are also required. In applications where high hardness, wear resistance, strength and good heat properties are required, high speed steels (HSS) are widely used today. HSS has high carbon content (> 0.4 wt.-%) and are therefore considered as unweldable, hence very limited effort has been made to manufacture HSS with SLM [4-7]. However, there is an increasing interest in producing crack-free HSS by SLM. In SLM, it is possible

possible to preheat the base plate to a few hundred degrees Celsius which has been used for HSS and proved successful. A few studies show that by preheating the base plate to 200°C it is possible to produce crack-free HSS with carbon contents of 0.8-0.9 wt.-% [5, 8, 9] and with an elevated temperature up to 500°C, HSS with even higher carbon contents of 1-1.3 wt.-% can be produced. In the SLM, the material solidifies at a high cooling rate (up to 10^6 K/s). The large temperature gradient between new deposited layer and previously formed substrate induces grains growing directionally, which in turn can cause brittleness. Besides the repetitive thermal cycles generate compression and expansion in the lattice, resulting in rather high internal stress, which in turn makes the component prone to cracking. By preheating the base plate, the temperature gradient is diminished, and thus the internal stress as well as the risk of cracking is reduced [5].

In this study, the properties of SLM printed high speed steel PEARL Micro®2012 with a carbon content of 0.61 wt.-% have been revealed and compared to those of a forged and rolled PM-HIP counterpart ASP®2012.

2. Materials and Methods

The materials used in this study are SLM produced PEARL Micro®2012 and forged and rolled PM-HIP ASP®2012. ASP®2012 is a high speed steel for hot- and cold-work applications where high toughness is needed. The composition of these grades is shown in Table 1. The powder materials were produced in Erasteel's large scale gas atomization plant. The optimal heat treatment of the PM-HIP material, to achieve the best combination of hardness and toughness is firstly soft annealing at 880°C followed by slow cooling, hardening in protective atmosphere at 975-1075°C followed by quenching and then tempering three times at 560°C for one hour [10].

Table 1. Chemical composition in wt.-% of the materials studied.

	C	Si	Cr	Mn	Mo	W	V	Fe
SLM	0.61	1.06	4.01	0.26	1.91	2.02	1.50	Bal
PM HIP	0.61	1.05	4.25	0.32	2.10	2.15	1.58	Bal

The powder for the SLM printed material was sieved to a fraction 15-63 µm. The flowability of the powder was characterized with a Hall flow meter according to ISO 4490, the apparent density according to ISO 3923-1, the tap density according to ISO 3953 and the particle size distribution with laser diffraction (Malvern Mastersizer 2000) according to ISO 13320.

Using the SLM powder, tetragonal specimens were printed in argon atmosphere with SLM®125 machine, and the standard operation parameters for H13 tool steel in the database of SLM® solutions were employed in the present work, see Table 2. The base plate was heated to 200°C. Two printing rounds were performed where the powder was recycled from the first round and used in the second round. The samples from the first round were removed from the building plate with wire Electrode Discharge Machining (EDM) in as-printed state. The samples from the second round were heat treated with stress relieving treatment, 650°C for 5

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hours, slow cooling down to 500°C and self-cooling to 25°C before removing them from the building plate using wire EDM.

Table 2. Standard H13 material parameters used for the printing in SLM125.

Material		Layer thickness		Core KPV				Border KPV			
H13		0.03 mm		Laser power (W)	Scan speed (mm/s)	Hatch distance (mm)	Focus (mm)	Laser power (W)	Scan speed (mm/s)	Hatch distance (mm)	Focus (mm)
				175	720	0.12	0	100	400	0.09	0
Filling KPV				Down Skin KPV				Up Skin KPV			
Laser power (W)	Scan speed (mm/s)	Hatch distance (mm)	Focus (mm)	Laser power (W)	Scan speed (mm/s)	Hatch distance (mm)	Focus (mm)	Laser power (W)	Scan speed (mm/s)	Hatch distance (mm)	Focus (mm)
150	450	0.08	0	100	1000	0.06	0	300	400	0.18	-8

The PM HIP powder was sieved to <500 µm, filled in a cylindrical capsule and HIPed. After HIPing to full density, the material was forged and hot rolled to Ø16 mm bars.

The surface roughness on the top and side of the printed samples was measured using a profilometer. One of the as-printed samples was tempered three times at 560°C for one hour each and one of the stress relieved samples together with a sample from the forged and rolled bar was soft annealed at 880°C, hardened at 1025°C and tempered three times at 560°C.

A hardness profile was measured from the top to the bottom on one as-printed and one stress relieved sample using Vickers indenter with 1 kg load. The Vickers hardness was also measured with 10 kg load on as-printed and heat treated samples. The microstructures were studied in a Light Optical Microscope (LOM) and a Scanning Electron Microscope (SEM) after etching in 4% Nital.

3. Results and discussion

The powder properties of the AM powder characterized before the printing are presented in Table 3 and the as-printed and stress relieved square samples are seen in Figure 1. A first visual study of the samples showed no clear defects or cracks in the samples and the arithmetic mean roughness value (Ra) for top and side was measured to 3.6 µm and 4.8 µm respectively.

Table 3. Results from the powder characterizations.

Powder characterization		
Flowability Hall flow meter	Flow time (s/50g)	18.6
Density	Apparent density (g/ cm^3)	4.2
	Tap density (g/cm^3)	5.0
Particle size distribution	d_{10} (µm)	19.5
	d_{50} (µm)	38.7
	d_{90} (µm)	70.8

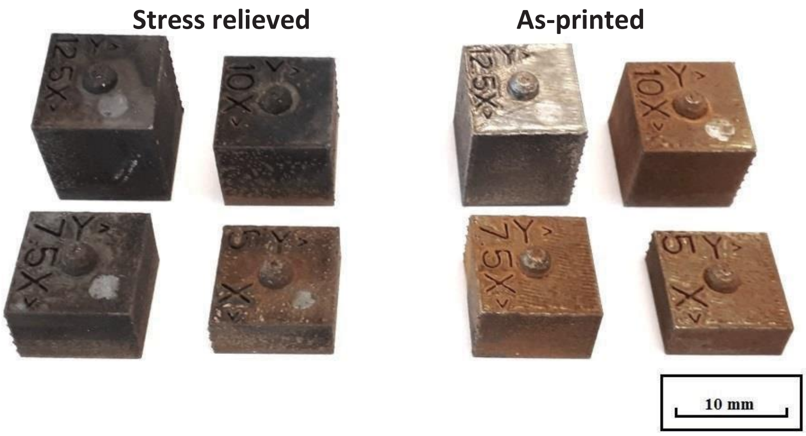

Fig 1. Stress relieved and as-printed square samples with heights between 5-12.5 mm.

In Figure 2, the hardness profiles measured in the building direction of as-printed and stress relieved square samples with a thickness of 10 mm can be seen. There are significantly greater hardness variations in the as-printed sample than in the sample that has been stress relieved. The hardness is also in average 100 HV1 higher in the as-printed state.

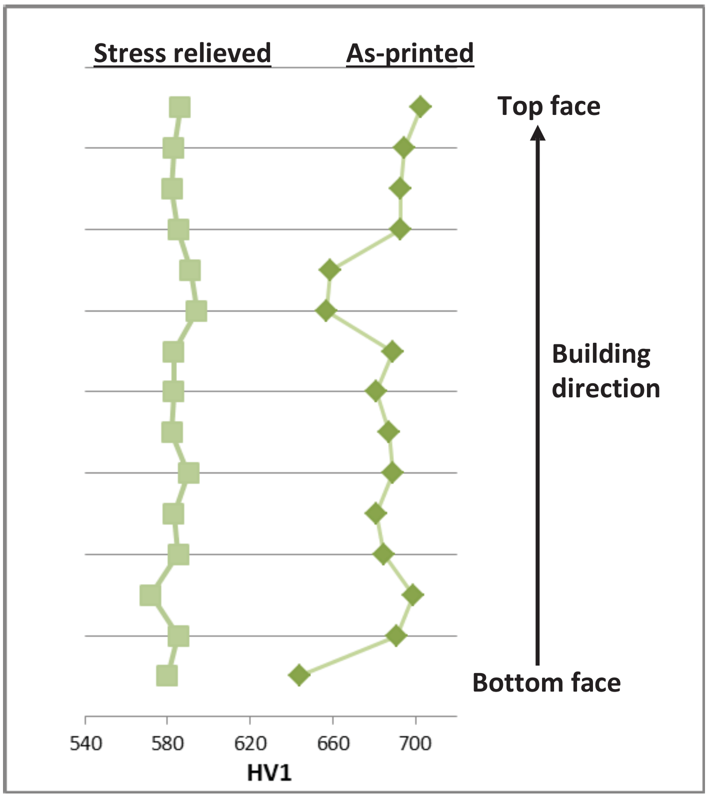

Fig 2. Hardness profiles measured from top to bottom face in the building direction of as-printed and stress relieved samples.

The fact that there is a difference in hardness between the as-printed and the stress relieved samples is also seen in the Vickers hardness measurements with 10 kg load presented in Table 4. There are also the hardnesses measured after further heat treatments of the as-printed and stress relieved samples as well as for the heat treated PM -HIP material presented. After tempering, the hardness of the as-printed sample increased with 67 HV10. The stress relieved sample achieved the same hardness as the PM-HIP material after being subjected to the same heat treatment, showing that the printed material has the same response to the heat treatment that PM-HIP material has in terms of hardness.

Table 4. Vickers hardness with 10 kg load (HV10) measured on the as-printed and heat treated samples.

Material	HV10
As-printed	683
Stress relieved	571
As printed, tempered	750
Stress relieved, soft annealed, hardened and tempered	593
PM-HIP, soft annealed, hardened and tempered	594

Figure 3 shows the LOM images of the microstructures in the etched cross sections of the as-printed and heat treated materials, and Figure 4 shows SEM images of the same etched microstructures taken with secondary electron detector. In the as-printed state, the laser scanning paths are visible where the hatch spacing and how the direction has been rotated between subsequent layers perpendicular to the construction direction can be seen (Figure 3a). In parallel with the building directions, the different layers that have been built up and the melt pools are seen instead (Figure 3b). One crack was also seen which appeared to have started at the edge. In LOM the microstructure appears to consist of a mixture of a dark and a bright area. At higher magnification, it is seen that the dark area consists of mixture of needle like martensitic structure and retained austenite (Figure 3c) and the bright area consists of a columnar dendritic and cellular structure (Figure 3d) which is even more evident in SEM (Figure 4a). The columnar dendritic structure crosses multiple melt pool boundaries and indicates epitaxial grain growth due to the temperature gradient that occurs during building. For the stress relieved material, smaller grains extended in the building direction with very distinct dark grain boundaries are visible (Figure 3e). The SEM image shows that the grain boundaries contain much precipitates and have taken a lot of etching, which is a typical appearance for a high speed steel tempered at a temperature above 600°C (Figure 4b). In the background, it is also possible to see residues of the columnar dendritic structure. The 3x560°C tempered as-printed material has obtained a more even structure, which, however, still consists of a needle like martensitic structure but which is mixed with both tempered martensite and a small amount of bright retained austenite (Figure 3f). The structure still contains some residues of the columnar dendritic structure and cellular structure (Figure 4c). The soft annealed, hardened and tempered printed material (Figure 3g) has a similar microstructure to the soft annealed, hardened and tempered PM-HIP material (Figure 3h) in LOM which contains a tempered martensitic matrix and small round carbides. The only difference in the printed material is that it is still possible to see some residues of the columnar dendritic structure. In SEM, the carbides are seen as very small bright spots (Figure 4d). In the heat treated PM-HIP material are also these very small carbides seen, but there are also

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somewhat larger carbides that are not visible in the printed material (Figure 4e). Otherwise, the martensitic matrixes have quite the same appearance.

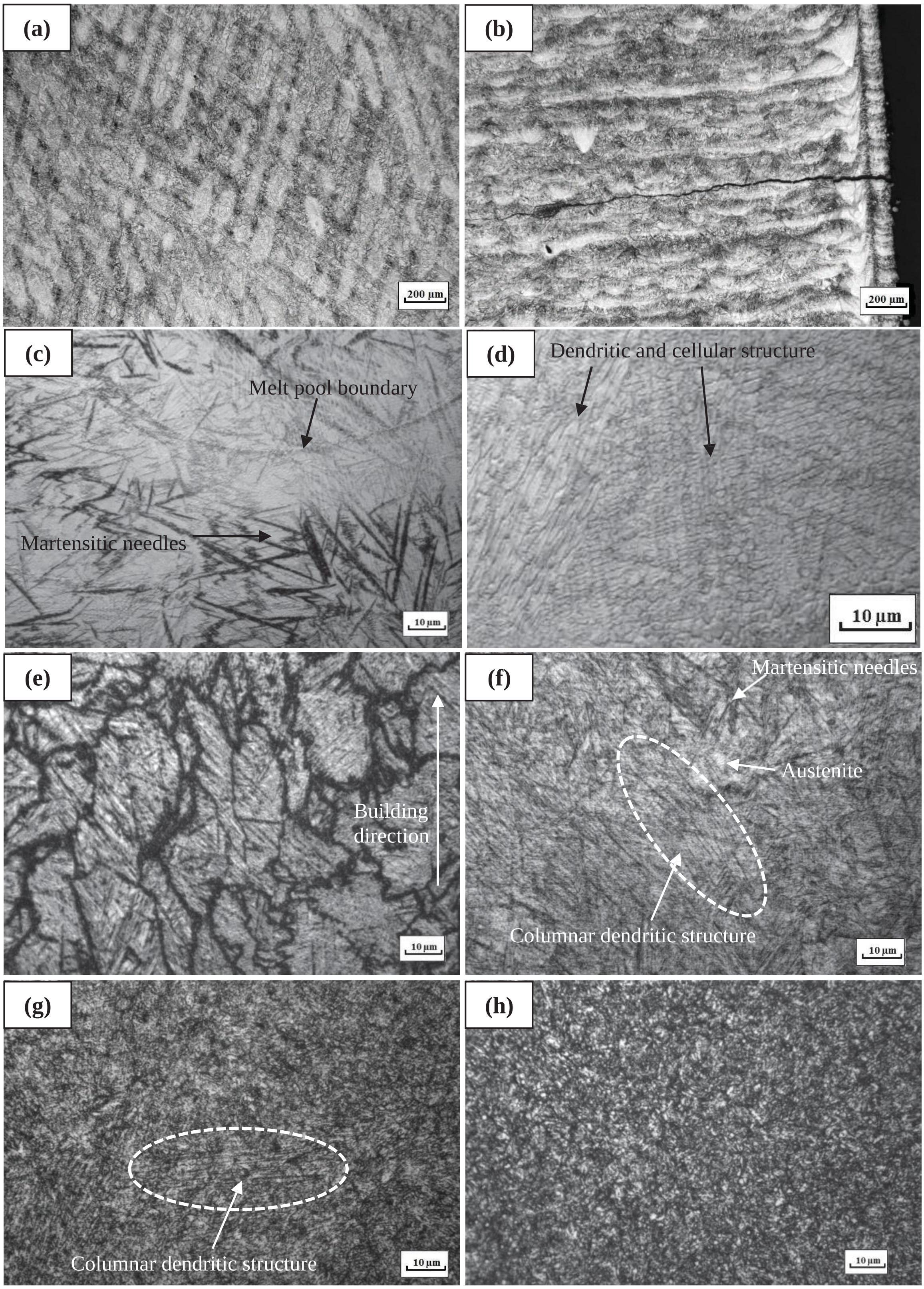

Fig 3. LOM images of etched cross sections of (a) as-printed material perpendicular to building direction, (b-d) as-printed material parallel to building direction, (e) stress relieved material parallel to building direction, (f) tempered as-printed material parallel

to building direction, (g) soft annealed, hardened and tempered stress relieved material parallel to building directions and (h) soft annealed, hardened and tempered PM-HIP material.

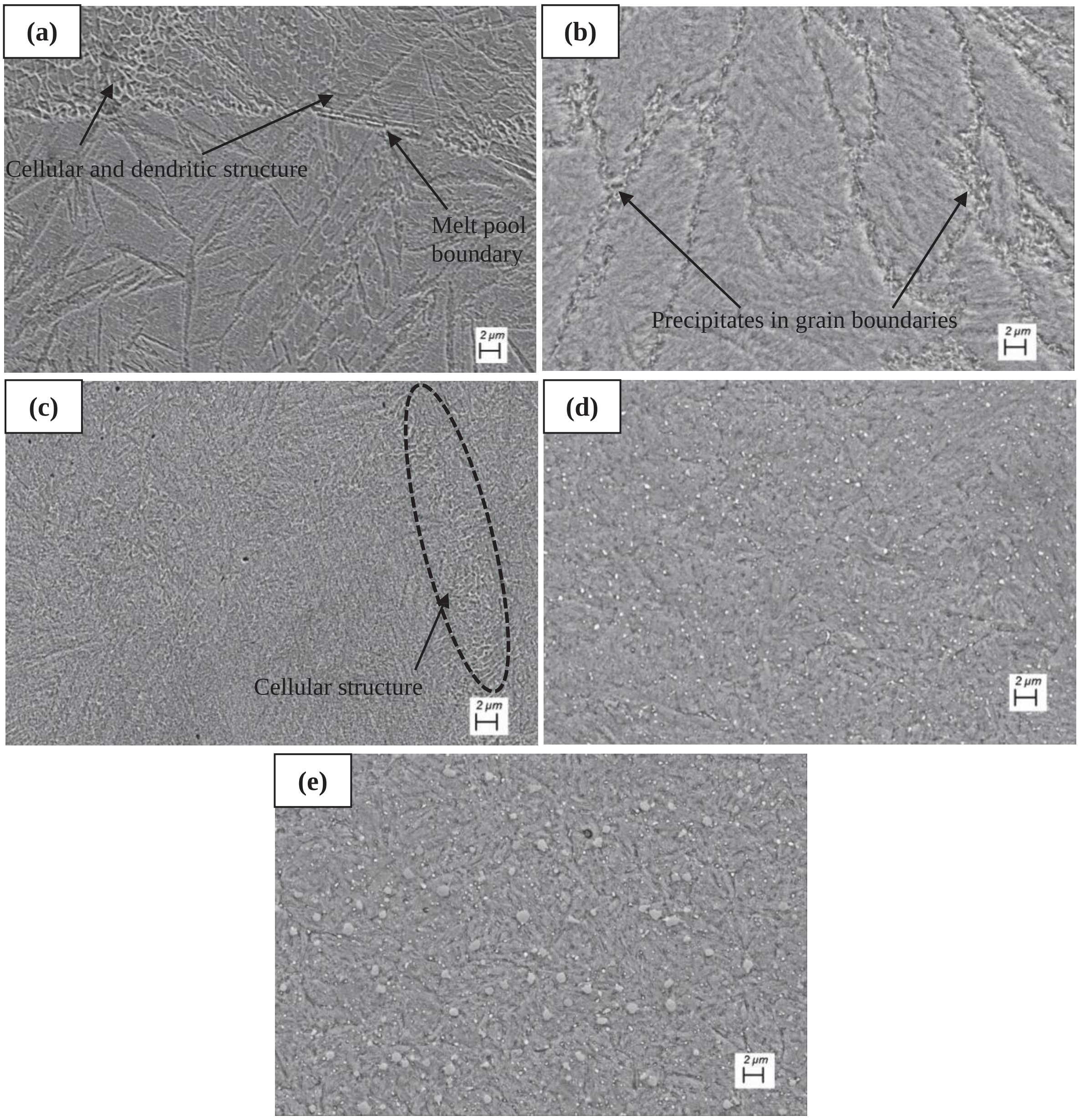

Fig 4. SEM images of etched cross sections of (a) as-printed material, (b) stress relieved material, (c) tempered as-printed material, (d) soft annealed, hardened and tempered stress relieved material and (e) soft annealed, hardened and tempered PM-HIP material.

Apart from the single large crack in the as-printed material (Figure 3b), generally no larger cracks could be found in any of the other cross section samples. On the other hand, very small cracks were commonly seen on the top surface of most SLM produced samples and some minor pores in the materials.

The fact that the printed specimen contains very few cracks does not mean that the material can be used to produce parts in the SLM without the right post treatment. High speed steels are generally very brittle and must be heat treated correctly to obtain optimum mechanical properties. If the material consists of retained austenite, untempered martensite and martensitic needles, the material is much more brittle and more sensitive to the smallest defects. The structures of the as-printed material, the stress relieved material and the tempered as-printed material contained just that and would therefore probably have a very low toughness and may fail if used in a tool or component under high mechanical stresses. The soft annealed, hardened and tempered printed material had similar microstructural appearance to the PM-HIP material containing tempered martensite and carbides. This makes it more possible to achieve similar mechanical properties such as the PM-HIP material. However, it is very important, just as for normally produced high speed steel that all possible crack initiation points and small cracks in the surface should be removed from components in order to achieve good mechanical properties.

4. Conclusions

In this study, the high speed steel PEARL Micro®2012 with a carbon content of 0.61 wt.-% has been produced with base plate preheating in a SLM 3D printer, using standard operation H13 tool steel parameters from SLM® solution. The hardness and microstructures have been reviewed after different heat treatments and compared to microstructures and hardness of a forged and rolled PM-HIP high speed steel counterpart ASP®2012.

The as-printed material achieved a hardness of 683 HV10 and a microstructure consisting of a mix of columnar dendritic and cellular structure, needle like martensitic structure and retained austenite. After tempering, the hardness increased to 760 HV10 and the microstructure still consisted of needle like martensitic structure but mixed with both tempered martensite and a small amount of retained austenite. Based on the microstructures, the materials are not estimated to be capable of achieving good mechanical properties due to the brittleness of untempered martensite and retained austenite.

When soft annealing, hardening and tempering the printed material as for the PM-HIP material, similar hardnesses of 593-594 HV10 and microstructures consisting of tempered martensite and small round carbides were obtained. It shows that there is a possibility that similar mechanical properties of SLM produced material can be achieved as of PM-HIP material. However, there was one large crack and some smaller cracks in the cross sections that were studied which must be avoided and/or removed for not impairing the mechanical properties.

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References

[1] S. Hoeges, et al., Development of a maraging steel powder for additive manufacturing, GKN sinter metals engineering GmbH, Hoeganaes Corporation.
[2] Gibson, I., Rosen, D. W., and Stucker, B., Additive Manufacturing Technologies: 3D Printing, Rapid Prototyping, and Direct Digital manufacturing, 2nd ed., Springer, New York, (2015), pp. 1–498.
[3] D. Herzog, et al., Additive manufacturing of metals, Acta Materialia (2016, Volume 117), p.371-392.
[4] J. Saewe, et al., Feasibility investigation for laser powder bed fusion of high-speed steels, Euro PM, Bilbao (2018).
[5] J. Saewe, et al., Feasibility investigation for laser powder bed fusion of high-speed steels AISI M50 with base plate preheating system, , BHM Berg- und Huttenmännische Monatshefte (2019, Volume 164), p.101-107.
[6] J. Sander, et al,. Microstructure and properties of FeCrMoVC tool steel produced by selective laser melting, Material and Design (2016, Volume 89), p.335-341.
[7] J. Kunz, et al., Influence of powder bed temperature on microstructure and post treatment of high speed steel AISI M50 processed by laser powder bed fusion, Euro PM, Bilbao (2018).
[8] K. Kempen, et al., Selective laser melting of crack-free high density M2 high speed steel parts by baseplate preheating, Journal of materials engineering and performance (2014, Volume 136), p.61026.
[9] Z.H. Liu, et al., A preliminary investigation on selevtive laser melting of M2 high speed steel, 5th international conference on advanced research and rapid prototyping, Leiria (2011).
[10] Erasteel, ASP2012 datasheet (2019, Volume 3).