Use of HIP Process in Post-Processing of Components Manufactured by SLM Technology from Magnetically Soft FeSi6.5 Powder

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Abstract. This paper presents the influence of the Hot Isostatic Pressing (HIP) process on the structure and properties of the components printed by Selective Laser Melting (SLM) technology. The samples were manufactured from magnetically soft FeSi6.5 alloy powder, using different SLM printing parameters. Semi-finished products were densified in the HIP process by varying its duration and isostatic pressure value. Density, hardness, and microstructure were tested on the printed parts both before and after HIP densification. The application of the hot isostatic pressure densification process indicates a change in its final properties concerning the feedstock components. The influence of variable SLM printing parameters of semi-finished parts on the production quality and properties after the HIP process was also observed.

Introduction

Constant technological development generates a demand for new components dedicated to electrical devices. Their efficiency, cost and effectiveness largely depend on the materials used in their construction. Considering, among others, electric motors, transformers, generators, or induction filters, the improvement of highly efficient magnetic materials is of particular importance [1-3]. One of them, which is part of the construction of modern electrical devices and can visibly improve their efficiency, is the Fe-Si alloy, which is classified as soft magnetic material. Due to high electrical resistivity, very low magnetostriction and low magnetocrystalline anisotropy, it has a high potential of being applied in new electrical devices [4]. Unfortunately, due to its fragility, it is challenging to manufacture finished parts with complex shapes by conventional methods. Currently, the progress in Additive Manufacturing technologies allows for the production of ready-made elements using various additive technologies without the need to conduct complicated and energy-consuming processing [2,4].

In the SLM (Selective Laser Melting) process, stresses arise in the printed element (e.g., thermal shock), which may cause their disintegration (e.g., cracking and crumbling). For this purpose, heat treatment is often used to remove stresses in the material and, consequently, improve the magnetic properties [4]. An important factor influencing the mentioned properties is the appropriate structure of the material. For this purpose, additional HIP treatment was performed on the elements printed with the SLM technique. The research presented in the article is a preliminary assessment of the possibility of producing soft magnetic materials FeSi6.5 using a combination of additive technologies and hot isostatic pressing. Materials intended for research in this work were manufactured using the SLM technology and various configurations of printing parameters [5]. Furthermore, the article presents an assessment of the impact of parameters (pressure, time) of post-process treatment by HIP on printed elements made of FeSi6.5 alloy made in various strategies (laser power, scanning speed) of SLM printing.

Research methodology and material

The samples were prepared using SLM 125 device from SLM Solutions. The printed samples were post-processing processed using HIP AIP8-30H device from American Isostatic Press, Inc.

The powder morphology was examined on a JEOL JXA-8230 microscope. The density was determined by the Archimedes method, using ethyl alcohol with a chemical purity of 99.98%. HV hardness was determined using a load of 1 kgf for 15 seconds. Microstructural studies were performed on the Olympus GX71F metallographic microscope and the Zeiss Evo MA10 scanning electron microscope. Phase testing was performed on the Seifert-FPM XRD7 device using the characteristic Cu Kα X-rays and the Ni filter as well as Seifert and Match software and ICDD PDF-4 + catalogue data from 2021.

The samples were prepared using SLM technology from FeSi6.5 alloy powder with a particle size below 100 µm (Fig. 1a). The powder was purchased from PMCtec GmbH (Precision Materials Chemicals and Technology). Examination of its morphology shows the presence of a mixture of globular and spherical particles. Before its use, the alloy powder was subjected to the process of removing moisture by isothermal annealing at the temperature of 120°C for 3 hours and to sieving to obtain the 20 - 63 µm fraction.

(a) (b)

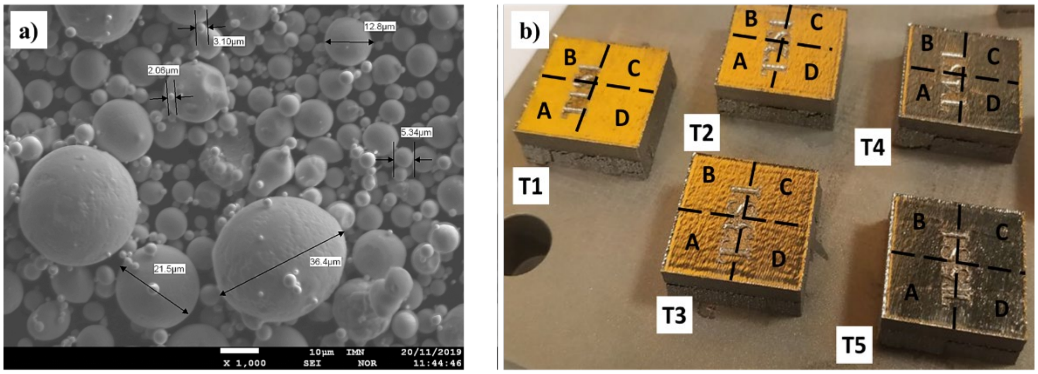


Figure 1. (a) SEM morphology image of the FeSi6.5 powder with particle size below 100 µm, (b) Photo of samples immediately after the printing process with visible support material made of 316L steel with marking of the sample cutting pattern

The samples were produced using five printing strategies, marking them sequentially T1, T2, T3, T4, and T5. The applied printing parameters and their values, along with the assigned markings, are summarized in Table 1. To avoid the printed elements' oxidation, the printing process was carried out in an argon atmosphere. The printing platform was heated to the temperature of 200°C to minimize the phenomena of thermal shocks and their influence on the initiation of possible cracks or delamination in the material. The produced printed elements were in the form of cuboidal samples with dimensions of 20 x 20 x 5 mm (Fig. 1b).

Table 1. Printing parameters assigned to the strategy

Strategy		T1	T2	T3	T4	T5
Laser power	[W]	180	200	220	200	220
Scanning speed	[mm/s]	600	667	733	571	629
Hatch distance	[mm]	0.12				
Layer thickness	[mm]	0.03				
Layer cooling time	[s]	15				

Each of the printed elements was cut into four parts and marked as A, B, C, and D (Fig. 1b). Samples with the A marking were considered as reference material samples, while the remaining samples were intended for the hot isostatic pressing process. HV1 density and microhardness were measured on all samples, both before and after the HIP process. The marking of the tested samples along with the parameters used in the hot isostatic pressing process are summarized in Table 2.

Table 2. Results of density and hardness measurements together with applied HIP process parameters

Sample		T1			T2			T3			T4			T5		
		1B	1C	1D	2B	2C	2D	3B	3C	3D	4B	4C	4D	5B	5C	5D
Before HIP	Density [g/cm^3]	7.46 ± 0.02			7.48 ± 0.01			7.46 ± 0.05			7.47 ± 0.03			7.47 ± 0.03		
	Hardness HV1	428 ± 19			424 ± 11			411 ± 15			404 ± 14			431 ± 16		
HIP parameters	Temperature [°C]	1200														
	Pressure [MPa]	100	200		100	200		100	200		100	200		100	200	
	Time [s]	240		480	240		480	240		480	240		480	240		480
After HIP	Density [g/cm^3]	7.46	7.47	7.47	7.47	7.46	7.46	7.47	7.46	7.47	7.48	7.46	7.46	7.48	7.46	7.46
	Hardness HV1	399	400	401	409	398	400	402	392	401	392	400	393	398	399	404

The course of the Hot Isostatic Pressing process corresponded to the pre-set parameters, which is illustrated by the temperature and isostatic pressure curves (Fig. 2). In all cases, the pressure was applied simultaneously with increasing temperature. The hot isostatic pressing process was carried out at different values of isostatic pressure (100 MPa, 200 MPa) and the duration of the process (240 and 480 minutes).

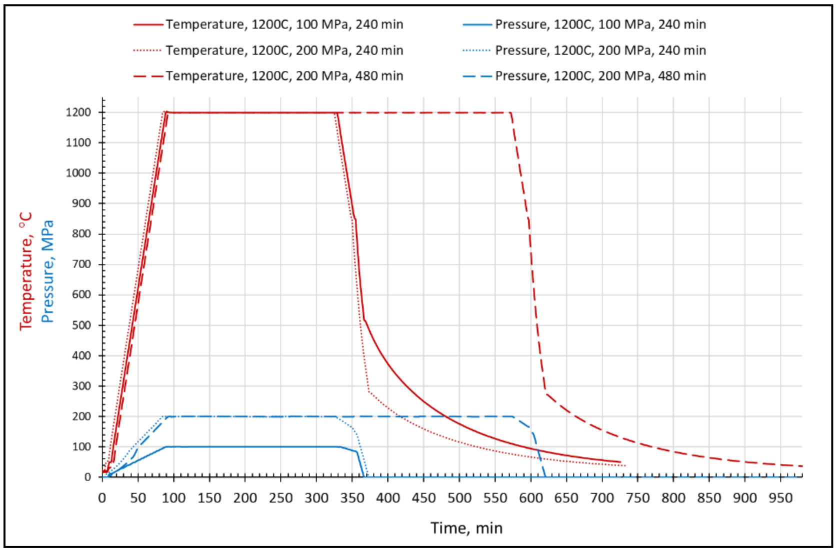


Figure 2. Course of the Hot Isostatic Pressing process

Due to the extensive microstructural research, the article presents the results for the maximum parameters of the HIP process, namely for the parameters: 1200°C, 200 MPa, and 480 min.

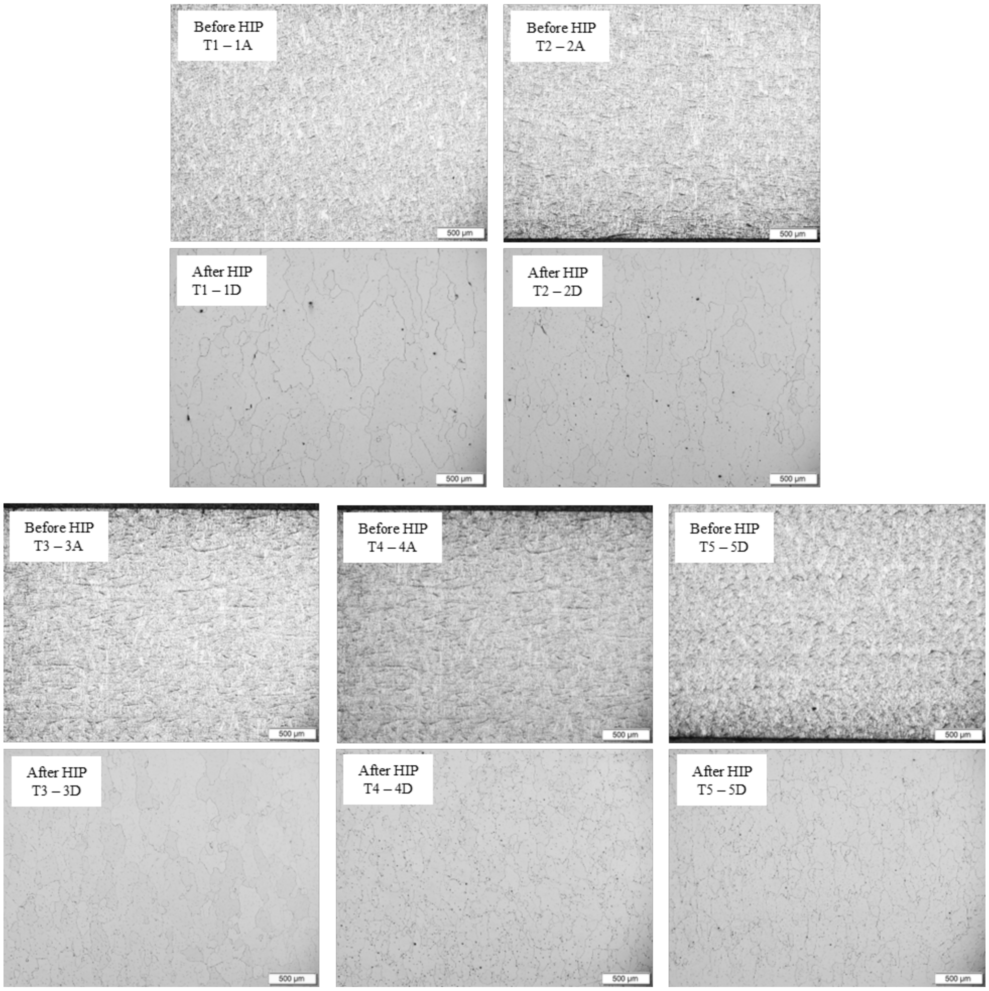
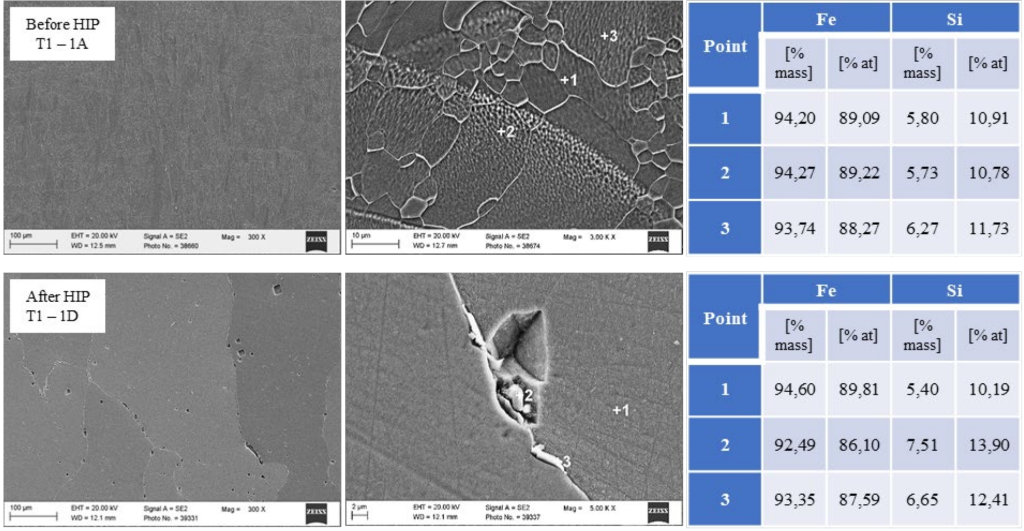


Figure 3. Samples microstructure, optical microscope, 1200°C, 200 MPa, 480 min



Point	Fe		Si	
	[% mass]	[% at]	[% mass]	[% at]
1	94,20	89,09	5,80	10,91
2	94,27	89,22	5,73	10,78
3	93,74	88,27	6,27	11,73

Point	Fe		Si	
	[% mass]	[% at]	[% mass]	[% at]
1	94,60	89,81	5,40	10,19
2	92,49	86,10	7,51	13,90
3	93,35	87,59	6,65	12,41

Figure 4. Microstructure of the samples, SEM

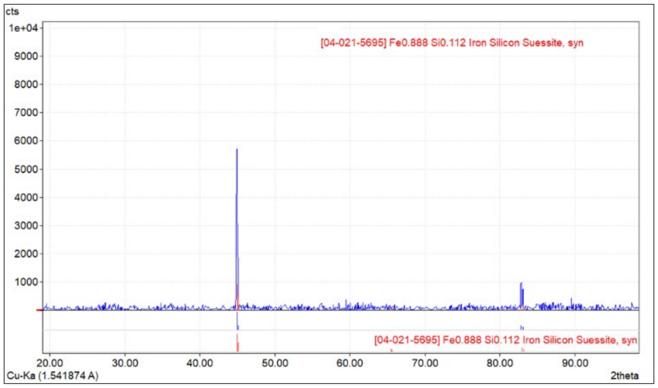


Figure 5. Phase composition of the sample after the HIP process, 1D (T1)

Discussion of the results

The analysis of the hardness and density measurement of samples after the SLM printing process shows the impact of changes in printing parameters (mainly laser power and speed) on the properties of the obtained semi-finished products. The highest density of 7.48 g / cm^3 was obtained for the sample with the T2 printing strategy, the lowest for the T1 and T3 strategies. The hardness of the printed elements was in the range of 404 - 431 HV1 (T4 and T5, respectively). Examination of the microstructure in all cases indicates the presence of irregular grains elongated in the direction of the laser beam movement (Figs. 3, 4).

After Hot Isostatic Pressing treatment, in most cases, a change in the density of prints in the range of ± 0.02 g / cm^3 (in relation to their value after SLM) was noticed depending on the SLM printing strategy and the parameters of the HIP process used. For all variants of the Hot Isostatic Pressing process (B - 1200°C, 100 MPa, 240 min; C - 1200°C, 200 MPa, 240 min; D - 1200°C, 200 MPa, 480 min), a decrease in hardness was noted, the highest by 7.66% for the sample marked with 5B, and the lowest for the sample marked with 4C by 0.99%.

Examination of the microstructure after the HIP process shows the presence of grains of different sizes depending on the printing strategy, the largest for the samples with the 1D and 2D numbers, the smallest for the samples with the 4D and 5D markings (Fig. 3). The SEM-EDS analysis showed the presence of light precipitates with increased silicon content up to 7.51 wt.%. (13.90 at. %) inside and at the grain boundaries, which, according to earlier work [6], can be related to the formation of Si-rich amorphous precipitates (Fig. 4).

Examination of the phase composition of samples after the HIP process shows the presence of a single-phase material with the chemical atomic composition of Fe - 88.8% at. and Si - 11.2% at. (Fig. 5).

The investigated microstructures show the presence of pores which, due to their regular shape, were probably formed during the preparation of samples for microstructural tests due to bright silicon-rich precipitates removed from the matrix material.

Observed herein changes in the microstructure generated by the post-treatment using the HIP method should also influence the magnetic properties of printed samples. Accordingly, optimization of HIP parameters in the context of magnetic properties will be a subject of further research.

Conclusions

As part of the research, the following conclusions were formulated:

1. The use of the Hot Isostatic Pressing process in the form of post-process treatment affects the microstructure's homogenization and improvement of printed elements' properties in SLM technology.
2. The quality of the elements obtained in the SLM printing technology impacts their

properties after using the Hot Isostatic Pressing process.

3. The combination and optimization of the SLM printing technology and the HIP process in producing FeSi6.5 magnetic soft materials has a positive effect on their properties and generates new opportunities for their use in producing elements with complex shapes.

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