Magnetic, electric properties and hardness of 17-4 PH stainless steel fabricated by selective laser melting

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Abstract. This research examines 17-4 PH stainless steel (SS) samples fabricated by selective laser melting (SLM). The samples were built in vertical direction, and it was necessary to load powder three times. This made the process suspended and led to the changes in temperature conditions. Despite a continuity of the samples, the samples were divided into three parts shared by visible interfaces. The samples were examined by optical microscopy and X-ray diffraction (XRD) analysis. The XRD data showed variations in phase compositions over the samples height. Magnetic and electric properties, hardness and microhardness of the samples in each parts were determined before and after the heat treatment (650 °C for 1 hour, cooling in a furnace). Obtained results show an inhomogeneity of properties over the samples height, but heat treatment led these properties to be uniformly distributed.

1. Introduction

The selective laser melting (SLM) is one of the 3D printing techniques. The SLM is used to obtain complex shape products directly. Scientists are interested in products properties, fabricated by SLM. Nowadays, numerous works are devoted to the study of mechanical properties and microstructure of as-fabricated products [1-3]. The SLM technique is widely used due to a number of its advantages: as-build parts of complex shape, re-using of removed powder, ability of producing components without post-processing, reducing of processing time and so on. The applications of SLM in different field of industries and medicine have been discussed in [4]. However, many issues related to the technological features of SLM process and properties of as-fabricated products remain unsolved.

The fabrication is carried out in the building chamber in inert atmosphere to protect the heated metal against oxidation. The SLM components are built by selectively melting powders within and between layers by a focused laser beam. The SLM component properties are influenced by process parameters. Process parameters, such as laser power, scanning speed, hatch spacing, layer thickness and building chamber atmosphere are determinative in SLM.

Most of the SLM research are devoted to three types of metals: iron, titanium, and nickel. Different variations of steel have also been investigated for processing by SLM. The 17-4 PH is a chromium-nickel-copper precipitation hardening stainless steel used for applications in marine environments,
power plants, and chemical industries because of its good mechanical properties and corrosion resistance. The cost steel has been already studied [6], but SLM steel has not been investigated enough. The study [1] examined the microstructure of 17-4 PH SS (commonly known as European 1.4542 and German X5CrNiCuNb16-4) samples obtained by powder atomized in nitrogen and argon atmospheres. The fabrication of such samples was carried out in one step.

If the size of the component is large, it is necessary to load the powder several times. This makes the process be suspended and leads to the changes in temperature conditions and causes the occurrence of interfaces between SLM parts. Such interfaces can have a modified structure and exhibit properties different from those of the component as a whole, which affects the performance characteristics. This phenomenon must be studied well.

This work is devoted to the study of distribution of magnetic, electric properties, as well as hardness and microhardness of the 17-4 PH stainless steel samples over the samples height. The samples were fabricated by SLM vertically with additional loading of a powder. The studied samples have two interfaces caused by powder loading.

2. Materials and Methods
The study was carried out on the 17-4 PH stainless steel (having compositions of Cr 15-17.5%, Ni 3-5%, Cu 3-5%, Nb 0.15-0.45%, Si<1%, Mn<1%, Mo<0.5%, C <0.07%) samples and interfaces, fabricated by SLM.

Samples 8x8x300 mm³ in size were vertically synthesized from the 15-40 μm powder in argon atmosphere using an EOSINT M280 equipment. The 300 mm height samples required three powder loads during fabrication. This caused the appearance of three parts separated by two interfaces.

It is well known that the SLM parts properties depend on process parameters and CAD laser strategy. In this study, the process parameters were as follows:
- scanning speed, 1000 mm/s;
- laser power, 195 W;
- laser spot, 40 μm;
- hatch spacing, 100 μm;
- stripes width, 10 mm;
- stripes overlap, 50 μm.

In this study, as shown in figure 1a, the laser beam was driven first through the shell, then, through the core. The core was melted by stripes. Stripes were rotated in each second layer by 900 relative to the layer below.

In figure 1b part 1 corresponds to the sample growth beginning, the part 2 is middle, part 3 is the farthest from the substrate plate. The occurrence of the interfaces was caused by suspension of the melting process when the additional powder was required.

The SLM parts structure was examined by light (optical) microscopy (OM) and X-ray diffraction (XRD) analysis before the study of properties and heat treatment. The study of the structure was carried out by Micromed MET OM. The XRD spectra of Cu (Kα) radiation of as-build parts were measured by DRON-3 system. The spectra were processed by PowderCell 2.4 Software.

1 – stripes width; 2 – stripes overlap; 3 – laser beam trajectory

Figure 1. Concept of SLM process (a) and sample side view (b).
The density values of each part were measured by Archimedes method. The magnetic and electric properties, as well as hardness and microhardness were measured for three parts of the sample. The properties were studied before and after the heat treatment (HT) (650 °C for 1 hour, cooling in a furnace). This post-processing allowed stress-relieving.

The magnetic properties of the samples were measured by a Remagraph C-500 developed by Magnet-Physik Dr. Streingroever GmbH (magnetization measurement error is less than 2%, induction measurement error is less than 1%). The electric resistivity was measured by a four-probe method (measurement error is 1%). The microhardness was measured using a Nanotest 600 (Micromaterials Ltd) equipped with a Vickers diamond pyramid with a load of 1 N and the dwell time of 5 seconds. The measurements were carried out for each parts and interfaces. Each part was subjected to 10 indentations, and interfaces, to 30 indentations. The results were averaged.

3. Results and Discussion

3.1. Microstructure
Figure 2-4 show micrographs of laser-melted EOS 17-4 PH SS sample showing fully remelted, dense structure at different magnifications. It can be seen that there are some differences in the shape and size of pores in samples from different parts of the SLM product. This phenomenon has been investigated in [5].

![Figure 2](image1.png)  
Figure 2. Part 1 microstructure at different magnifications obtained by Micromed MET

![Figure 3](image2.png)  
Figure 3. Interface 1-2 microstructure at different magnifications obtained by Micromed MET
3.2. Density and Porosity
The parts 1-3 were examined by Archimedes method. The part 1 exhibited density of 7.63 (relative density 97.8%), part 2 – 7.70 (relative density 98.7%), part 3 – 7.79 g/cm³ (relative density 99.9 %). This means that the maximum of voids concentration was observed in the part 1. These voids are associated with powder properties and process parameters. It is known that powders size distribution, laser power, and laser speed influence on a porosity of SLM fabricated parts. The obtained porosity is less than 3%. This means that process parameters were chosen well.

3.3. XRD analyses
Figure 5 shows XRD spectra for parts 1, and 3. According to the XRD data, phase compositions were different in parts 1-3. The results showed that the level of fcc phase varied from 20 to 33 % as well as bcc phase varied from 80 to 67 % over the sample height. According to the study [1] results, the SLM samples melted in Ar-atmosphere are principally martensitic (and ferromagnetic). In the study [1] it was confirmed by transmission electron microscopy images, which showed lath martensite oriented to the building direction.
3.4. Magnetic Properties

Figure 6 shows the hysteresis loops for three parts of the as-built sample before a heat treatment (a) and after a heat treatment (b). Obtained hysteresis loops show the uniformly distribution of magnetic properties over the sample height before a heat treatment. This distribution is connected with powder loading.

![Figure 6](attachment:figure6.png)

Figure 6. Parts 1-3 hysteresis loops before (a) and after a heat treatment (b).

Figure 6a shows the hysteresis loops for three sample parts before the heat treatment. The remanence and saturation magnetization values of the parts 1-3 were found to be different over the sample height, i.e., the remanence varied from 0.20 T (part 1) to 0.11 T (part 3) and saturation magnetization varied from 485 kA/m (part 3) to 651 kA/m (part 1). The coercive force varied insignificantly.

Figure 6b shows the hysteresis loops for three sample parts after the heat treatment (650 °C for 1 hour, cooling in a furnace). The heat treatment changes the magnetic properties over the sample height. Figure 7 shows the difference between hysteresis loops before and after the heat treatment.

![Figure 7](attachment:figure7.png)

Figure 7. The comparison of hysteresis loops of part 3 before and after the heat treatment

Table 1 summarizes obtained data about magnetic properties before and after the heat treatment.
Table 1. Magnetic properties of SLM sample.

| Heat treatment | $M_s$, kA/m | $B_r$, T | $H_C$, kA/m |
|----------------|-------------|----------|------------|
| Part 1         |             |          |            |
| before HT      | 651         | 0.20     | 3.8        |
| after HT       | 968         | 0.43     | 4.1        |
| Part 2         |             |          |            |
| before HT      | 582         | 0.16     | 3.8        |
| after HT       | 953         | 0.42     | 4.3        |
| Part 3         |             |          |            |
| before HT      | 485         | 0.11     | 3.9        |
| after HT       | 936         | 0.39     | 4.3        |

According to the table 1 data, the heat treatment caused remanence and saturation magnetization uniformly distributing over the sample height. After the heat treatment, the saturation magnetization and remanence increased, i.e. part 3 saturation magnetisation increased from 485 to 936 kA/m, and remanence increased from 0.11 to 0.39 T. This changes may be associated with phase transformations occurred after the heat treatment, for example, with a $\gamma$(fcc)-$\alpha$(bcc) transformation.

3.5. Electric Properties

The electric resistivity measurements of parts 1-3 were carried out before and after the heat treatment. Table 2 summarizes obtained results.

Table 2. Electric properties of SLM sample.

| Heat treatment | $\rho$, $\mu$Ohm-cm |
|----------------|---------------------|
|                | Part 1  | Part 2  | Part 3  | Cast Steel |
| before HT      | 90      | 89      | 86      | 98         |
| after HT       | 83      | 84      | 86      | 86         |

The results of measurement show that heat treatment caused an electrical properties uniformly distributing over the sample height. Table 2 contains a cast steel electric resistivity value (reference data). These values before and after heat treatment are in a good agreement with obtained result for SLM parts.

3.6. Hardness and Microhardness

Parts 1, 2, and 3 exhibited hardness of 34.9, 34.4, and 31.8 HRC, respectively. After heat treatment, the hardness increased by 17%. This fact is related to fcc Cu-rich particles precipitation within the dislocation tangles. The difference between the hardness values of parts 1-3 was reduced after the heat treatment.

The cast steel hardness value (reference data) varies from 33 to 39 HRC before the heat treatment and from 35 to 42 HRC after heat treatment. This data is in a good agreement with the obtained values. Parts 1, 2, and 3 exhibited the microhardness of 307, 287, and 373 HV, respectively. One of the interfaces was found to have microhardness as high as 687 HV, which suggested the local metal hardening.
4. Conclusion
This work is devoted to the magnetic and electric properties, hardness and microhardness of the sample fabricated by the SLM in three steps.

The samples of 300 mm high were melted in vertical direction. The powder was loaded three times during fabrication. This made the process suspended and led to the changes in temperature conditions. Despite a continuity of the samples, the samples were divided into three parts shared by visible interfaces.

The results of this research showed that the magnetic, electric properties, as well as hardness and microhardness were not homogeneous when the samples were melted vertically. This fact was associated with inhomogeneous phase composition over the sample height. The XRD analyses data confirmed this fact.

The heat treatment caused hardness, remanence and saturation magnetization increasing and uniformly distributing over the sample height as well as stress revealing.

According to the hardness and remanence changes, the $\gamma$(fcc)-$\alpha$(bcc) transformations have been occurred. The hardness growth may be also related to fcc Cu-rich particles precipitation within the dislocation tangles.

This work gave an important result. This result must be taken into account during fabrication of high samples.

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