The effect of heat treatment on the structure and mechanical properties of additively manufactured stainless steels

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Abstract. Selective laser sintering was applied for stainless steel samples additive manufacturing. It was found that in the as fabricated state, the samples consist of martensite and residual austenite, contained significant dislocation density and residual stresses. Additively manufactured samples had an ultrafine-grained structure with predominantly high angle grain boundaries and were free of crystallographic texture and residual porosity. The ultimate tensile strength of the as-built steel was 960 MPa. Annealing caused a decrease in residual austenite, but did not significantly affect the size and morphology of the grains. The ultimate strength after annealing increased markedly while maintaining plasticity.

1. Introduction

Selective laser sintering (SLS) at the moment is one of the most promising methods of additive production. This is associated with its ability to facile making of complex three-dimensional geometries with quite moderate manufacturing effort without interjacent operations of thermomechanical treatment. It is well known that set of the final product properties including its mechanical characteristics and physicochemical properties is defined by the microstructural features formed under SLS, which, in turn can be affected by varying such parameters as initial powder size and quality, fusion modes and some others conditions implied during SLS process [1,2].

A major drawback of the SLS process is the presence of impurities and porosity accompanied by the structure heterogeneity leading to degradation of the final product properties. Studies aimed to improve both the structural characteristics and mechanical properties of steel samples obtained by SLS is a relevant issue due to the fact that steel is the key material in different industrial applications [1-3].

The allotropy of Fe-C alloys is an important issue which can promote new insightful ways of governing the structure parameters during SLS process in the presence of significant temperature gradients in the fusion zone. For instance, the retained austenite present in martensite-aging steels after quenching at high cooling rates can significantly affect on the overall complex of properties of SLS produced EOS PH1 steel.

This paper presents the results of study of microstructural and mechanical characteristics of the SLS produced samples of EOS PH1 martensite aging steel in as produced and annealed states.
One should note that the investigation of SLS produced steels counterparts is of particular importance for both fundamental material science and for practical applications due to the fact that the obtained results contribute to the development of new approaches aimed on improving the properties of existing products and can provide new insights for design of new manufacturing concepts.

2. Experimental
SLS processing of the powder of EOS stainless steel PH1 was done by EOSINT commercial equipment system. The processed samples were in the form of cylinders with a 10 mm diameter of and a 30 mm height. The chemical composition of the powder corresponds to the DIN 1.4540 compositions (table 1).

Table 1. Chemical composition of EOS Stainless Steel PH1 (wt.%).

|   | Fe   | Cr    | Ni    | Cu  | Mn | Si | Mo | N  |
|---|------|-------|-------|-----|----|----|----|----|
|   | balance | 14-15.5 | 3.5-5.5 | 2.5-4.5 | ≤1 | ≤1 | ≤1 | 0.15-0.45 |

Microstructural analysis of the samples processed by SLS was performed using a scanning electron microscope (SEM) Tescan Mira 3LMH using the back-scattered electrons (BSE) and secondary electrons (SE) detectors. XRD phase analysis of the studied samples was conducted on a DRON-4-07 diffractometer using Cu-Kα radiation. Tescan VEGA 3 SBH SEM with an energy dispersive X-ray spectroscopy (EDS) attachment was used for the chemical composition analysis. Electron backscattered diffraction (EBSD) investigations were realized using the Tescan Mira 3 LMH SEM. Small grains comprising three or fewer pixels were automatically not considered in the analysis in order to improve the reliability of the EBSD data. 2° lower limit boundary misorientation cut off was used for exclusion of orientation noise induced spurious boundaries. Differentiation of low-angle and high-angle boundaries was based on A 15° criterion. The grain size was estimated by determining each grain area and its circle-equivalent diameter calculation, the method is also known as grain reconstruction [4].

Mechanical properties were investigated by means of tensile tests on the specimens with a cross-section of 1.9×0.4 mm² and a length of 4 mm. The tests were conducted at ambient temperature and a nominal strain rate of 10⁻³ s⁻¹ on the Instron 5982 testing machine.

The microhardness of the additively manufactured samples in the as-processed state, as well as after heat treatment, was measured on the cross section of cylindrical samples along the diameter with a step of 1 mm on a Vickers microhardness tester (MKV-h21) for 15 s at an applied load of 100 gf.

3. Results and discussion
XRD analysis revealed the initial powder was composed of 90% martensite phase (bcc) and 10% austenite phase (fcc) which is probably due to high rate of quenching during the preparation of powder. In the additively manufactured sample, XRD analysis (figure 1) revealed the absence of crystallographic texture, and phase composition, similar to initial structure consisting of 90% martensite and 10% austenite phase, increased lattice parameters values and high internal stresses which is the result of high cooling rates that are usually used during the SLS process.
Figure 1. X-ray diffraction pattern of Stainless Steel PH1 sample in the as fabricated state.

Figure 2a presents the results of microstructural analysis of the PH1 Stainless Steel sample processed by SLS in the longitudinal sections. The bulk monolithic structure without any visible microcracks and pores can be observed. Both grain size and the chemical composition of the samples revealed the high degree of homogeneity along the analyzed area. One could observe a large fraction of elongated grains, but determination of preferable elongation direction seems not to be possible in this case.

Since EOS Stainless Steel PH1 is characterized by having outstanding mechanical properties, specifically in the precipitation hardened state, some of the samples after SLS were heat treated at 480 °C for 1, 2, 3 and 4 hours. The white arrows in figure 2b point small dark intermetallic precipitates that appeared after hardening heat treatment.

The X-ray diffraction patterns exhibit 5% of the fcc austenite phase after annealing. Besides, the level of internal stresses and values of lattice parameters decreased significantly with increasing duration of annealing, while coherent scattering region increased.

EBSD mapping (figure 3) revealed mainly bcc and few fcc grains in the longitudinal and transverse sample sections. The average size of both martensite and austenite grains was estimated to be about 0.4 μm. Low and high angle boundaries (LAB) and (HAB) are shown by white and black lines respectively. The fraction of bcc phase HABs was about 65%, therewith 67% of them represent the boundaries between the variants that corresponds to the orientation relationship of Kurdyumov-Sachs.
This mechanism of martensitic transformation is a result of the phase instability due to high local stress fields present in the crystal lattice.

Figure 3. EBSD maps of SLS samples in the as fabricated state (a) and after annealing at 480 °C for 3 hours (b).

EBSD analysis allowed us to calculate the average grain size and the fraction of HABs and LABs in the samples under study (figure 4). It is necessary to note that the grain size was calculated taking into account LAB. Annealing caused some increase in grain size and the amount of HABs, while the percentage of LABs decreased (figure 4b).

Figure 4. Dependence of the grain size $d$ (a) and fractions $\%$ of HAB (——) and LAB (-----) (b) on the duration of annealing at 480 °C.

Martensitic aging steels are highly alloyed steels with a low carbon content. They are known for possessing superior strength and toughness without losing ductility. A low carbon content helps to prevent thermal cracking upon cooling [5]. The main strengthening of this type of steels occurs during aging heat treatments, causing precipitation of intermetallic compounds. According the data sheet of EOS Stainless Steel PH1, an aging temperature of 480 °C was chosen.

Figure 5 indicates the evolution of microhardness (HV) as a result of aging. It is obvious that HV in the as processed sample is about 300 HV with some elevated values on the edges of the cross section that may be related to the cooling rate on the side surface of the sample during SLS.
Hv
1 hour
2 hours
3 hours
4 hours
as built

-4 -3 -2 -1 0 1 2 3 4 R, mm

Figure 5. Distribution of microhardness along the diameter over the cross section in the as-received state and after precipitation annealing at 480 °C for 1, 2, 3 and 4 hours.

Annealing at 480 °C led to a substantial (almost 30%) increase in microhardness. At the same time, the duration of annealing practically did not affect the value of microhardness - all the microhardness distribution curves after annealing are grouped at the same level about 450 HV.

The results of tensile tests at room temperature are summarized in table 2.

| Structural state  | YS, MPa | UTS, MPa | Elong., % |
|-------------------|---------|----------|-----------|
| As built          | 925     | 960      | 21        |
| Aging time 1 hour | 1195    | 1240     | 16        |
| Aging time 2 hours| 1160    | 1190     | 18        |
| Aging time 3 hours| 1190    | 1240     | 15        |
| Aging time 4 hours| 1080    | 1210     | 18        |

It is obvious that the yield strength (σy) as well as tensile strength (σt) increased substantially after aging, while the elongation value (δ) decreased. The strain hardening induced by heat treatment can be attributed to the precipitation of intermetallic compounds in the form of small particles, well distinguishable in the BSE images (figure 2b). It can also be observed that EOS Stainless Steel PH1 show a rapid aging response. During the first hour the strength and the hardness increase to maximum values.

The obtained values of tensile strength for SLS samples are close to those for samples of the same composition produced by rolling and subjected to the same thermal treatments [6].

4. Summary
The reported results provide a strong evidence that the additive manufacturing treatment of PH1 Stainless Steel by means of SLS-processing results in formation of an ultrafine grained material with predominantly high angle grain boundaries and free of residual porosity and crystallographic texture. High temperature gradients present during SLS process are responsible for formation of non-equilibrium microstructures in the as-fabricated state, e.g. retained austenite in martensite, nonequilibrium values of lattice parameters and significant residual stresses. Subsequent aging at 480°C raises rapidly strength and microhardness to maximum values during the first hour of heat
treatment. The mechanical properties of SLS samples are not worse than those of the samples obtained by conventional rolling

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