

Correlation between heat treatment process parameters, phase composition, texture, and mechanical properties of 12H18N10T stainless steel processed by selective laser melting

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Abstract. The phase composition, texturing degree, oriented stresses and strains of the crystal lattice, the physical and mechanical properties of austenitic steel 12H18N10T (AISI 321) manufactured by selective laser melting (SLM) before and after heat treatment with various temperatures and protective gas composition have been investigated. SLM stainless steel has better mechanical properties, given that the phase volume content is as follows: γ -Fe ~ 65%, α -Fe ~ 20%, NiTi ~ 5%; the texturing degree equals to $T_{111} \sim 0.70$ and the dominant crystal-lattice orientation is (111). Such a phase composition is formed in the steel after annealing at 620 °C in argon. As the temperature rises during the stabilizing annealing process up to 825 °C, the phase transformation $\gamma \rightarrow \alpha$ takes place in SLM steel with a change in the phase concentration γ -Fe and α -Fe in the range from 40 to 50%. In the high-temperature intermetallic phase NiTi, dissolution at 825 °C and a multiple rise of the FeO concentration lead to a change in the crystal lattice parameter of the γ -Fe and α -Fe phases and a decrease in the mechanical properties of SLM steel. This study showed that the improvement of mechanical properties was due to the separation of high-temperature intermetallic phase from austenite and an increase in the concentration of iron carbide Fe_3C in SLM steel.

1. Introduction

Selective Laser Melting allows manufacturing components of virtually any complexity, obtaining high performances of composite materials. The application of austenitic stainless steel powders in additive technologies, in particular, SLM, is of utmost interest both for research and application needs. Numerous studies have revealed a variety of features in the structure and properties of the laser melted austenitic steel powder. Despite the large number of various studies on stainless steels manufactured by SLM, not much focus is given to the influence of heat treatment on their structure and properties [2]. Sedlaka and Rican (2015), Hussein and Hao (2013), Wang and Yang (2013) determined that the above mentioned steels do not have a phase transformation $\gamma \leftrightarrow \alpha$ [1] over a wide range of temperatures and cooling rates. This paper aims to investigate the effect of heat treatment process parameters on the phase composition, internal stresses, texturing degree, as well as the physical and mechanical properties of the 12H18N10T item manufactured by SLM.

2. Materials and Experimental Procedure

Austenitic steel 12H18N10T (equivalent to AISI 321) was chosen as the subject of this study. Table 1 shows its chemical composition.



Table 1. Chemical composition of austenitic steel 12H18N10T, wt.%.

Fe	C	Cr	Ni	Ti	Si	Mn	P	S
Balance	0.093	17.67	9.59	0.55	0.70	0,95	0.06	0.009

SLM of powder from steel 12H18N10T was performed under a protective atmosphere of nitrogen on the Concept LaserM2 Cusing Machine (Germany). The size of SLM manufactured samples in the first batch was 10x10x15 mm. The dimensions of the second batch of test samples were in accordance with GOST 1497-84. Annealing of the samples from steel 12H18N10T manufactured by SLM was performed in a KP-110A Furnace and stabilization annealing - in a MonoThermHK.669.N.6gr Vacuum Furnace by ALD. The heat treatment (HT) process parameters are shown in table 2.

Table 2. HT process parameters of SLM manufactured samples from 12H18N10T (air-cooling).

Specimen pos.no.	HT type	Temperature, °C	Time at temperature, h.	Environment during HT
0	No heat treatment	-	-	-
1	Annealing	620	2	Air
2				Argon
3	Stabilization annealing	825	2	Argon
4				Vacuum

Phase composition, structure and strained condition of the samples from the first batch were studied by X-ray diffractometry.

The studies were conducted on a Bruker D8 Advance Diffractometer. The diffraction analysis parameters were as follows: voltage 50 kW, power 1000 μ A (1mA), copper anode with wavelength of $K\alpha_1=0,154055\mu\text{m}$. Diffraction patterns were processed in the DIFRAC.SUITE EVA software. The phase concentrations were determined by means of their volume content in accordance with the technique described in papers [3, 4]. The α -Fe and γ -Fe phase ratios was determined by the Rietveld method using Topas Software. The texturing degree T_{111} (T_{110}), the oriented stresses Δs and strains Δa of the crystal lattice, and the crystal lattice parameter change was specified in accordance with the techniques [5], [6], and [7], respectively. Static tests of the second batch samples were carried-out according to GOST 1479-84. Short-time strength tests of heat-treated samples were performed on the LFMZ-100 Machine.

3. Results

3.1 The influence of temperature values and medium during heat treatment on the phase composition, texture, stresses and strains in the crystal lattice of SLM steel

Steel 12H18N10T, manufactured layer-by-layer by SLM, before and after heat treatment, has a two-phase structure with a two-component texture. The structure of the SLM steel prior to heat treatment is γ -Fe with the crystal lattice parameter – $a=3.5853 \text{ \AA}$ and α -Fe with crystal lattice parameter – $a=2.8723 \text{ \AA}$ (table 3). Intermetallic compound FeNi, iron carbide Fe_3C and oxide FeO are also formed in SLM steel. The texturing degree $T_{111}=0.75$ shows that the dominant orientation of the crystal-lattice is (111). In the process of annealing at 620 °C in air or argon, the dominant phase is also austenite (FCC), dominated by the (111) crystal orientation (table 3, 4). The crystal lattice of SLM steel also does not change, which is confirmed by stability of the crystal lattice parameter. However, an increase in the α -phase content in the steel up to 29% (air) and 26% (argon) leads to a decrease in the texturing degree T_{111} by 7%. The minimum content of Cr_3C_2 was formed in SLM steel during air annealing. The formation of a high temperature intermetallic compound NiTi and an increase in the volume content of iron carbide Fe_3C were observed in SLM steel after annealing in an argon environment (table 4).

The phase transition $\gamma \leftrightarrow \alpha$ was not found in SLM steel after annealing at 620 °C. However, the $\gamma \rightarrow \alpha$ phase transition took place in SLM steel when rising the temperature during stabilizing annealing up to 825 °C (table 4). The concentration of the α -phase in the volume was raised up to 56%, which is two times higher than the similar parameter in SLM steel. The preferential crystallographic orientation from (111) changes to (110) (table 4). The volume content of FeO oxide is multiply increased. The high-temperature dissolution of the NiTi intermetallic alloy at 825 °C and a multiple increase in the FeO concentration lead to reduction of the crystal-lattice parameter of the γ -Fe and α -Fe phases. In this regard, we can conclude that Fe-dependent defects constitute the majority. The phase ratio of the α -Fe and γ -Fe phases is reversed when the stabilizing annealing medium changes from argon to vacuum (table 4). There is no essential difference in the T_{111} and T_{110} texturing degree of the α -Fe and γ -Fe phases (table 4). Negative and minimum oriented stresses in the crystal lattice of the γ - and α -phases indicate insignificant compression stresses.

Table 3. HT process parameters of SLM steel, crystal lattice parameter and oriented stresses of the crystal lattice.

	HT parameters			$a/\Delta a \cdot 10^{-3}, \text{\AA}$		Δs	
	$T, ^\circ\text{C}$	Environment	$T, \text{hrs.}$	(111) γ -Fe	(110) α -Fe	γ -Fe	α -Fe
No heat treatment	-	-	-	3.5853/1.6	2.8723/-2.1	-1.0038	-1.0035
Annealing	620	air	2	3.5853/1.6	2.8723/-2.1	-1.0043	-1.0040
Annealing	620	argon	2	3.5853/1.6	2.8723/-2.1	-1.0045	-1.0035
Stabilization annealing	825	argon	2	3.5818/2.6	2.8708/-1.5	-1.0028	-1.0029
Stabilization annealing	825	vacuum	2	3.5836/2.1	2.8708/-1.5	-1.0036	-1.0037

Table 4. Phase composition and structure characteristics of SLM steel.

Spec.	Volume ration of all possible phases in SLM steels, %							Phase weight ratio		Texturing degree	
Pos.no.	γ -Fe	α -Fe	Fe_3C	FeNi	FeO	NiTi	Cr_3C_2	γ -Fe	α -Fe	T_{111}	T_{110}
0	77.17	18.40	1.87	0.77	1.77	-	-	76	24	0.75	0.25
1	73.31	22.84	1.03	0.93	1.86	-	0.01	71	29	0.70	0.30
2	67.00	20.45	5.52	0.51	1.59	4.92	-	74	26	0.70	0.30
3	39.44	54.86	0.08	-	5.60	-	-	44	56	0.37	0.63
4	50.60	39.17	4.51	0.33	5.38	-	-	57	43	0.51	0.49

3.2 Effect of phase composition, texture, stresses and strains in the crystal lattice of SLM steel on its mechanical properties

Table 5 shows the mechanical properties of steel 12H18N10T manufactured by SLM before and after HT. Table 4 represents the properties of the cast steel 12H18N10T for the sake of comparison. Figure 1 shows the dependence of the mechanical properties of steel on the volume content of all incoming phases.

Table 5. Mechanical properties

Specimen No	UTS, MPa	0.2%YS, MPa	$\delta, \%$
Cast workpiece (GOST 5949-75)	510	196	40
0	651	481	47
1	667	488	46
2	679	487	42
3	660	446	56
4	638	429	51

It is found that steel 12H18N10T, manufactured layer-by-layer by the SLM process, has higher mechanical properties in comparison with cast steel and steel without HT. SLM steel has maximum values of 0.2% YS (0.2 % yield strength) and UTS (ultimate tensile strength) and a minimum value of tensile strain δ , similar to cast steel giving that its phase composition is as follows: γ -Fe \sim 65%, α -Fe \sim 20% and NiTi \sim 5%.

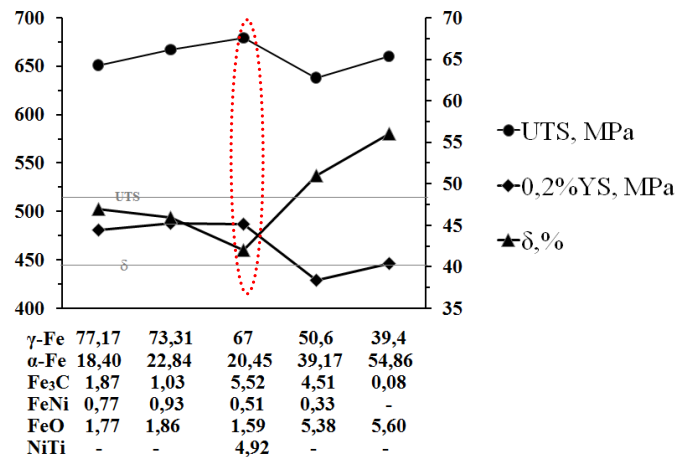


Figure 1. The dependence of mechanical properties of SLM steel

The texturing degree is $T_{111}=0.70$, and the preferential crystallographic orientation is (111). In case of an increase in the volume content of the γ -Fe phase and a decrease in the volume content of the α -Fe phase in SLM steel, its mechanical and tensile properties decline (figure 1, table 5). The limit 0.2% YS of SLM steel is 2.5 times higher than that of cast steel.

4. Conclusion

In this work, we studied the influence of the parameters of the heat treatment process on the phase composition, oriented strains of the crystal lattice, the texturing degree and the mechanical properties of steel 12H18N10T manufactured by SLM.

Steel 12H18N10T, manufactured by SLM, before and after HT, has a two-phase structure with a two-component texture. The phase transition $\gamma \leftrightarrow \alpha$ does not occur in a wide range of temperatures and cooling rates during the SLM process, as well as during the annealing process of SLM steel at 620 °C in the air or argon environment. The dominant phase retains the same, i.e. austenite (FCC), orientated in the (111) plane. However, the volume ratio of the Fe₃C iron carbide increases and the high temperature intermetallic compound NiTi is formed in SLM steel during annealing at 620 °C in argon.

Such a change in the phase composition results in a maximum increase in 0.2% YS up to 679 MPa, UTS up to 487 MPa and tensile strain δ up to 42%, which is close to the properties of cast steel. The volume content of incoming phases in SLM steel is: γ -Fe - 67%, α -Fe - 20.45%, NiTi - 4.92%, Fe₃C - 5.52%, FeNi - 0.51%, FeO - 1.59%.

Phase transformation $\gamma \rightarrow \alpha$ takes place in SLM Steel with an increase in temperature during stabilizing annealing up to 825 °C. The dissolution of the high-temperature intermetallic alloy NiTi at 825 °C and a multiple increase in the concentration of FeO lead to a decrease in the crystal lattice parameter of the γ -Fe and α -Fe phases and mechanical properties SLM steel.

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