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Microstructure and properties of laser surface alloyed PM austenitic stainless steel

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ABSTRACT

Purpose: The purpose of this paper is to analyse the effect of laser surface alloying with chromium on the microstructural changes and properties of vacuum sintered austenitic stainless steel type AISI 316L (EN 1.4404).

Design/methodology/approach: Surface modification of AISI 316L sintered austenitic stainless steel was carried out by laser surface alloying with chromium powder using high power diode laser (HPDL). The influence of laser alloying conditions, both laser beam power (between 0.7 and 2.0 kW) and powder feed rate (1.0-4.5 g/min) at constant scanning rate of 0.5m/min on the width of alloyed surface layer, penetration depth, microstructure evaluated by LOM, SEM x-ray analysis, surface roughness and microhardness were presented.

Findings: The microstructures of Cr laser alloyed surface consist of different zones, starting from the superficial zone rich in alloying powder particles embedded in the surface; these particles protrude from the surface and thus considerably increase the surface roughness. Next is alloyed zone enriched in alloying element where ferrite and austenite coexists. The following transient zone is located between properly alloyed material and the base metal and can be considered as a very narrow HAZ zone. The optimal microstructure homogeneity of Cr alloyed austenitic stainless steel was obtained for powder feed rate of 2.0 and 4.5 g/min and laser beam power of 1.4 kW and 2 kW.

Practical implications: Laser surface alloying can be an efficient method of surface layer modification of sintered stainless steel and by this way the surface chromium enrichment can produce microstructural changes affecting mechanical properties.

Originality/value: Application of high power diode laser can guarantee uniform heating of treated surface, thus uniform thermal cycle across treated area and uniform penetration depth of chromium alloyed surface layer.

Keywords: Laser surface alloying; High power diode laser; Stainless steel; Duplex microstructure

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1. Introduction

Laser surface alloying is a unique method where the external alloying elements in the form of powder, past, suspension, electrolytic coatings and plasma or flame sprayed coatings are introduced into a surface on the substrate, as preplaced addition material or injected directly into the weld pool, treated by means of a high power laser beam. Laser surface alloying of austenitic stainless steels is a common and widely used method of their surface modification. Laser alloying is able to achieve optimized surface quality while maintaining the bulk material's properties thus become the technique predisposed to achieve high local corrosion properties and wear resistance of commonly applied metallic materials, like stainless steels [1], tool steels [2], Ti alloys [3], Mg alloys [4], Al alloys [5] and many others.

Laser surface alloying is a well known technique used to improve mechanical properties and corrosion resistance of stainless steels surface layer. The comprehensive studies on the influence of surface laser treatment of various austenitic grades have shown the improvement of their intergranular [6], pitting [7] and cavitation erosion resistance [8] and also wear resistance after laser surface melting [9]. Laser surface remelting was successfully applied to restore localized corrosion resistance in sensitized [8] and in cold-worked and sensitised stainless steel [10]. Further improvement of surface layer in respect of wear resistance can be obtained by the alloying of stainless steel with hard particles such as carbides Cr_3C_2 [11] in aim to locally reinforce the surface of AISI 316 austenitic stainless steel. The surface hardening of austenitic stainless steel can be achieved with different routs i.e. the incorporation of hard particles of TiC, SiC, WC and carbon alloying [12] in order to form carbides as well as alloying with Si_3N_4 nitrides [13] and borides and their mixes [14] or elements such as Mo [15] resulting in the modification of their resistance to pitting or intergranular corrosion and stress corrosion cracking. The laser alloying of AISI 316 stainless steel with Al-Si powder also considerably improves cavitation erosion resistance and hardness [16].

The comprehensive studies of laser surface alloying of 316L with various both austenite and ferrite formers elements and their compounds were carried out by Kwok et al. [17]. The mixture of binder and Cr powder was painted on the stainless steel surface and then laser treated using a 1.1kW CW Nd-YAG laser. The authors in alloyed surface layer identified ferrite as the major phase, where the Cr content was 35-38% and superficial microhardness of 350HV.

The rapid melting and solidification during laser surface remelting produce fine grained microstructure as well as the homogenization of chromium distribution and dissolution of sulphide inclusions and carbides detrimental for corrosion resistance of stainless steels. The rapid cooling is a key factor influencing the formation of austenitic stainless steel microstructure that is extremely sensitive even to small changes in the cooling rate which may influence the solidification mode and thus influence microstructural changes and quantity of delta ferrite. The presence of delta ferrite depends also on the chemical composition of the alloy and commonly ranges from a few to over 10%. Depending on the alloy composition, the austenite present in microstructure can derive from the eutectic reaction and partial solid state transformation of ferrite or as a result of ferrite decomposition during post solidification cooling [18].

The austenitic 316L stainless steel is an important structural material for automotive parts, where the stainless steels are used for several applications like exhaust components, oxygen sensor bosses, ABS sensors and many other sintered parts [19]. Laser surface remelting of PM austenitic stainless steel may result in the improvement of overall material properties in surface layer due to density reduction and microstructure refinement, therefore the mechanical and corrosion performance will increase. Moreover, laser surface alloying with chromium may extra enrich the chemical composition thus enhance its properties and corrosion resistance and extend the range of possible applications. The porosity present in PM stainless steels affects thermal conductivity by acting as a thermal insulator, thus probably reducing the depth of penetration and slowing the rate of cooling comparing to wrought alloys and may also result in reduction of level of stress in the welded parts [20]. The laser surface alloying of single phase wrought austenitic stainless steel with ferrite forming element like Cr can produce duplex (austenite + ferrite) microstructure in surface layer thus increasing the hardness and corrosion resistance of processed surface [21].

Besides of numerous researches on LSA of wrought stainless steels there is still the lack of research on exact characterization of laser alloying with chromium, especially for sintered stainless steels. In present study PM stainless steel type AISI 316L was surface alloyed with Cr powder and the influence of laser alloying processing conditions on materials properties were evaluated.

2. Experimental procedure

2.1. Substrate material

The austenitic stainless steel powder type 316LHD (EN 1.4404) of Hoeganes with the nominal chemical composition supplied by the producer (Tab. 1) and particle size of <150µm was applied. The Acrawax was used as a lubricant in the quantity of 0.65 wt.%. Premixes were prepared in Turbula mixer for 20 min. and then uniaxially compacted in specimens of 10x10x55mm at 700MPa. The de-waxing process was performed at 550°C for 60 minutes in a nitrogen atmosphere. Samples were then sintered in a vacuum furnace with Ar backfilling at temperature 1250°C for 60 min. During the sintering cycle a solution annealing at 1050°C/1h was done and then the rapid cooling under pressure of 0.6MPa N_2 was applied with cooling velocity of 6ºC/s calculated in the range of 1250-400°C.

The sintered stainless steel 316L shows density of 7.2 g/cm^3 and open porosity of about 5% and ca. 92% of theoretical density. The microstructure of sintered stainless steel prior the laser treatment was composed of austenitic grains with presence of some twinned grains.

Table 1.
The nominal chemical composition of austenitic stainless steel 316LHD powder

Powder	Elements concentration, wt. %								
	Cr	Ni	Mo	Si	Mn	C	N	S	Fe
316LHD	16.2	12.3	2.2	0.26	0.1	0.019	0.05	0.006	bal.

2.2. Laser surface alloying

The laser surface alloying was done using Rofin DL 020 high power diode laser (HPDL) with rectangular laser beam spot at argon atmosphere with the following laser parameters: radiation wavelength 808±5nm, beam output power (continuous wave) 2300 W, beam focal length 82/32mm, laser beam spot dimension 1.8-6.8mm, power density range in the laser beam plane 0.8-36.5 kW/cm^2.

The alloyed surface layers on sintered stainless steel were produced as single stringer beads, the laser beam was focused on the top of specimens. The long side of laser beam spot was set perpendicularly to the alloying direction. The laser beam was guided along longer side (55mm) of specimens, the side compatible with their pressing direction. The surface of sintered stainless steel was laser alloyed with Cr powder (of particle grain size <300μm) that was injected directly into the weld pool in quantities of 1, 1.5, 2 and 4.5 g/min, respectively. The laser alloying was performed according to conditions presented in Table 2 at constant scanning rate and various laser beam power.

Table 2.
Conditions of the laser surface alloying process

Powder feed rate, g/min	Laser beam power, kW	Scanning rate, m/min
1.5	0.7	0.5
4.5		
1.0	1.4	
2.0		
4.5	2.0	

2.3. Materials characterization

Microstructure observations and geometrical characteristics of weld bead were carried out in the light optical microscope (LEICA ME F4M) and LEO 1450 VP scanning electron microscope (SEM) with the EDS probe.

The roughness of alloyed surface was evaluated using Surtronic 3+ of Taylor Hobson profilometer. The roughness parameter R_a was measured on the testing length of 4mm. The Vickers microhardness $HV_{0.05}$ was measured on the cross-section of alloyed surface in the FM-AKS 9000 of Future-Tech automated microhardness tester and a depth profile of the microhardness was determined.

The evaluation of phase composition was made using X-ray diffractometer with the filtered copper lamp rays at acceleration voltage of 45kV and heater current of 40mA. The measurements were made in diffraction angle 2θ from 40 to 100° of 2θ. The surface of samples was polished prior to x-ray analysis in aim to eliminate the influence of non finished surface layer. The phase quantity was estimated using Averbach and Cohen method of direct comparison assuming that $V_\gamma + V_\alpha=1$ [22].

3. Results and discussion

3.1. Geometrical characterization of alloyed surface layer

The dimensions of laser alloyed zone change with applied laser beam power and remain uninfluenced by powder feed rate during alloying, as presented on Figs. 1 and 2. The increase of the laser beam power resulted in widening of surface size of remelting zone, from about 6mm for 0.7 kW of laser beam power to about 7.2mm for 2.0 kW.

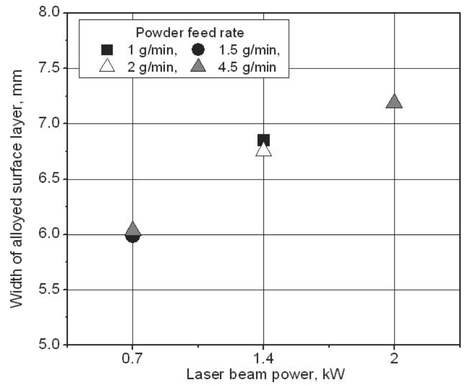

Fig. 1. The width of Cr alloyed surface layer of the sintered austenitic stainless steel 316L

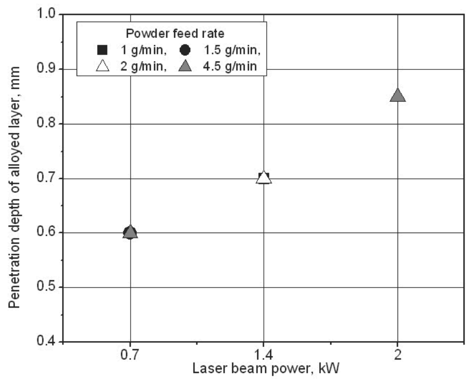

Fig. 2. The penetration depth of Cr alloyed surface layer of the 316L sintered austenitic stainless steel

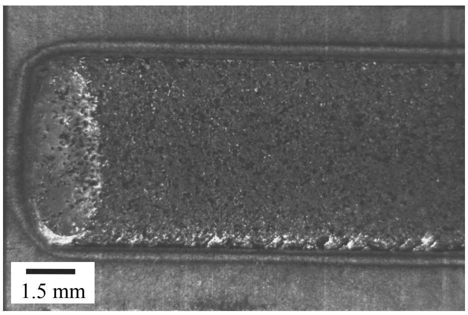

Fig. 3. The surface view of sintered 316L after laser alloying with Cr at laser beam power of 1.4kW and powder feed rate of 2.0g/min

The penetration depth of alloyed layer increased with the laser beam power and the maximal depth was equal 0.85mm for the highest laser power applied (Fig. 2). The authors previously

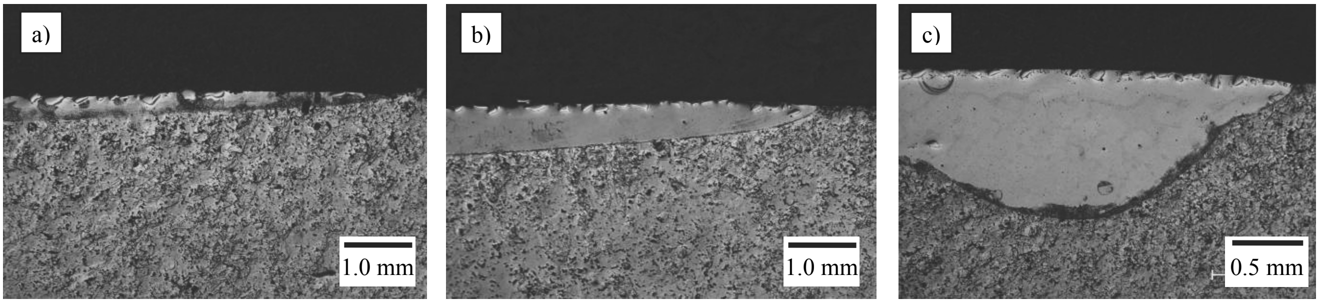

Fig. 4. The cross section of chromium laser alloyed 316L sintered stainless steel at increasing from a) to c) the laser beam power and powder feed rate, respectively: a) 0.7kW and 1.5g/min, b) 1.4kW and 2.0g/min and c) 2.0kW and 4.5g/min

reported in [23] that sintered austenitic stainless steel remelted in adequate conditions shows maximal penetration depth of 1.0mm, so the presence of additional alloying material in the form of powder decreased the maximal possible penetration depth of about 15%. Generally, sintered stainless steels exhibit lower penetration depth comparing to wrought ones due to porosity that retains heat and acts as an insulator and thus decreases thermal conductivity of material. The surface of alloyed layer of sintered 316L stainless steel has silver metallic shine (Fig. 3), and the weld bead of laser alloyed layer is uniform, flat and smooth without any visible undercuts or superficial cracks. The cross-sectional views of chromium alloyed surface of 316L sintered austenitic stainless steel are presented in Fig. 4.

The alloyed zone for lower laser beam powers - Figs. 4a and 4b show flat weld bead shape, while for alloying at 2.0kW it has a more regular oval shape.

The surface roughness expressed by Ra parameter increased when alloying with chromium was applied. The sintered stainless steel surface prior laser treatment shows roughness Ra=1.5μm and it strongly increases after laser alloying. The highest roughness Ra=7.3μm was registered for laser beam power of 1.4kW at powder feed rate of 2.0 g/min that was about twice of Ra=4.7μm measured for the maximal values of studied laser conditions (Fig. 5). The high surface roughness involve application of final surface finish treatment, such as grinding and polishing, but obtained penetration depth permit to realize such treatments without a risk of decreasing material properties.

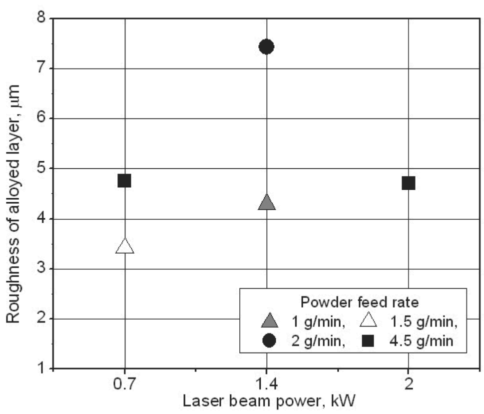

Fig. 5. The surface roughness of 316L sintered stainless steel after laser surface alloying

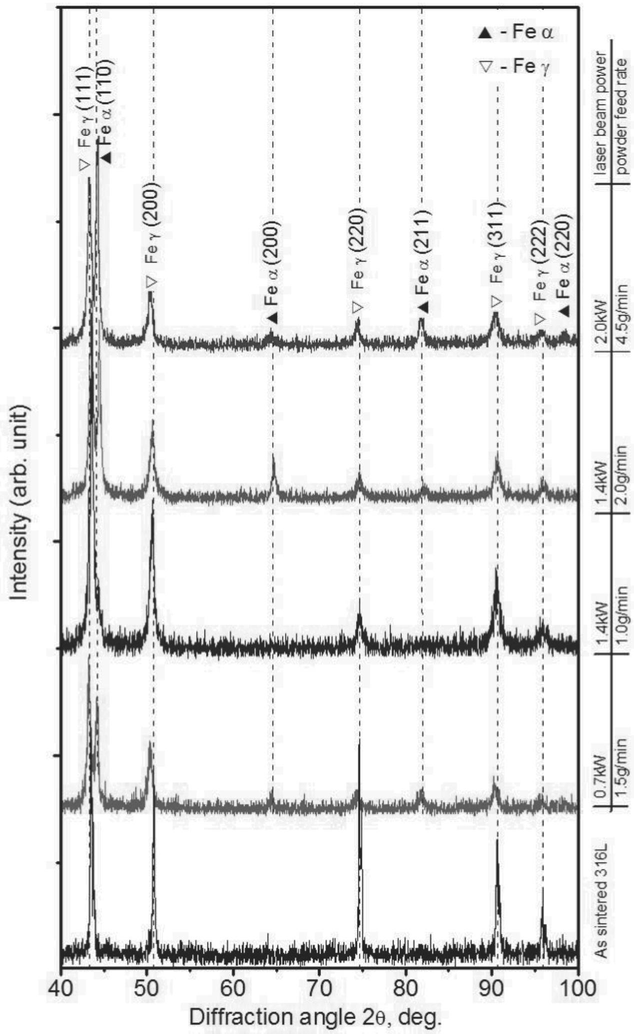

Fig. 6. The x-ray diffraction patterns of laser surface alloyed and as sintered stainless steel 316L

Prior to diffraction studies the alloyed surface was polished and overall depth of removed material was about 0.5mm, thus the embedded non dissolved chromium powder particles were removed. Polishing of samples eliminated the influence of non finished surface layer and the presence of pure chromium particles at the surface. The x-ray analysis was performed from the top of produced stringer bead surface.

The diffraction analysis of laser chromium alloyed 316L stainless steel confirmed presence of austenitic and ferritic phases (Fig. 6). The diffraction peaks origin of austenite Fe γ (111), (200), (220), (311), (222) and ferrite Fe α (110), (200), (211), (220) were clearly identified from x-ray patterns for all alloying conditions except the powder feed rate of 1.0g/min at laser beam power of 1.4kW. The strongest diffraction lines deriving from ferritic phase were registered for alloying conditions: laser beam power of 1.4kW and powder feed rate 2.0g/min and laser beam powder 2.0kW at maximal applied powder feed rate of 4.5g/min.

The increase of laser beam power and Cr powder feed rate resulted in increase of Feα peak intensity, thus the quantity of ferrite increased from 33% at laser beam power of 0.7kW and powder feed rate of 1.5g/min to 38% for laser powder of 2.0kW at maximal powder feed rate of 4.5g/min.

The metallographic studies revealed presence of different zones in alloyed surface. Starting from the top, the first one is the zone rich in alloying powder particles embedded in the surface; these particles protrude from the surface and thus considerably increase the surface roughness. The next zone is the alloyed zone enriched in alloying element.

The next zone can be considered as a transient zone between properly alloyed and the base metal. This zone shows slightly changed structure comparing to the substrate material and can be considered as a very narrow HAZ zone. The shape and depth of mentioned zones strictly depend on applied laser alloying conditions (scanning rate, laser beam power and particularly of chromium powder feed rate). The laser beam power at constant scanning speed influence the size area of surface layer where the microstructural changes take place. The laser beam power is also related to the shaping of remelting bottom and convexity of remelting face that are influenced by strong convection motions within the melted metal.

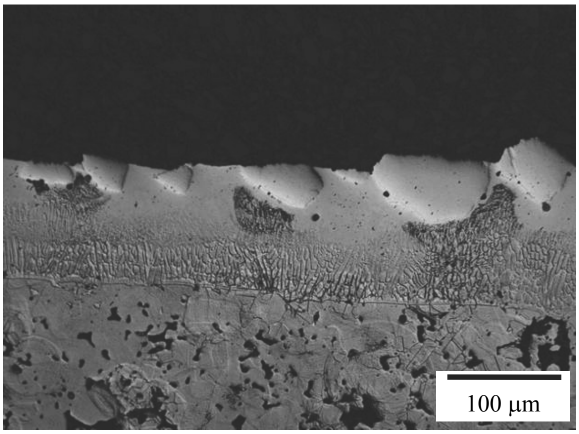

Fig. 7. The edge zone of 316L laser alloyed with Cr at laser beam power 0.7kW and powder feed rate of 1.5g/min

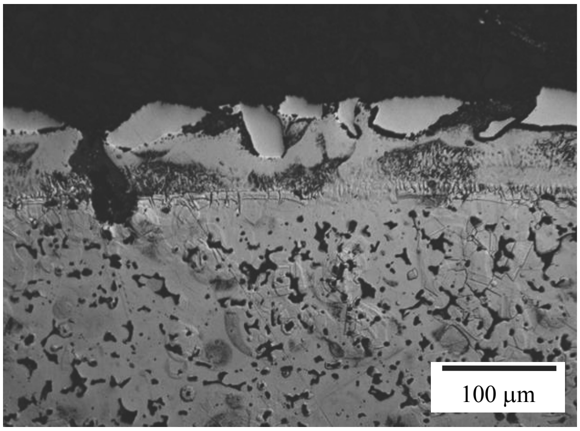

Fig. 8. The edge zone of 316L laser alloyed with Cr at laser beam power 1.4kW and powder feed rate of 2.0g/min

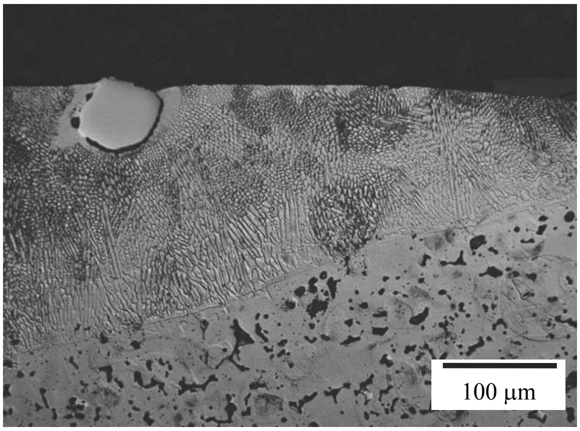

Fig. 9. The edge zone of 316L laser alloyed with Cr at laser beam power 1.4kW and powder feed rate of 1.0g/min

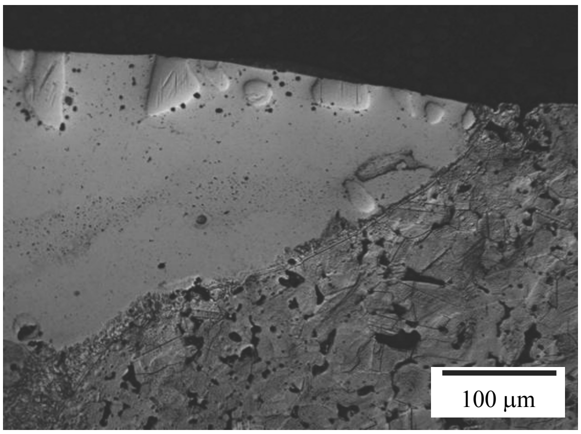

Fig. 10. The edge zone of 316L laser alloyed with Cr at laser beam power 2.0kW and powder feed rate of 4.5g/min

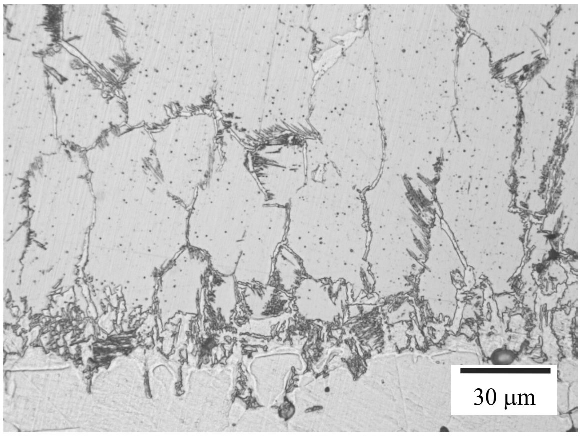

Fig. 11. The interface between alloyed zone (top), transient and base metal of 316L laser alloyed with Cr at laser beam power 2.0kW and powder feed rate of 4.5g/min

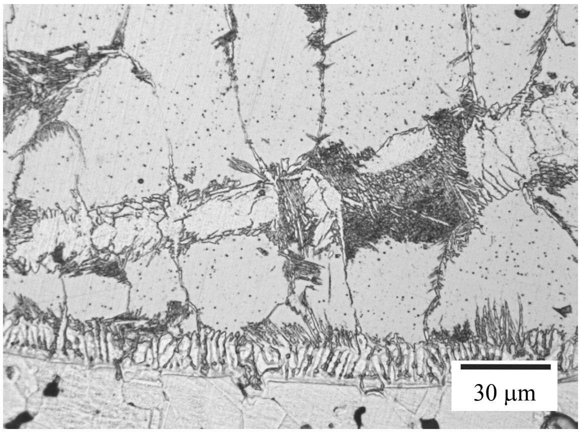

Fig. 12. The central zone of 316L laser alloyed with Cr at laser beam power 2.0kW and powder feed rate of 4.5g/min

Application of 0.7kW laser beam power for chromium alloying of 316L at powder feed rate of 1.5 and 4.5g/min is not sufficient to obtain uniform distribution of alloying powder in remelted zone. Figs. 7 and 8 present such remelting surfaces, where no dissolved chromium powder particles are embedded in steel surface. Between those particles the chromium enriched region can be seen and following zone where only remelting took place and microstructure shows cellular-dendritic morphology. The microsegregation of chromium at solidified cell boundaries results in the observed etching differences. The remelting zone, where the heat transfer proceeds in all directions shows fine grained cells of both rectangular and trapezoidal shape the cells form long parallel laths (Fig. 9). The initial porosity after laser surface alloying was completely eliminated.

Increasing the laser beam power to 1.4kW resulted in more intensive melting of alloying powder injected to molten pool. The chromium powder feed rate of 1.0 g/min is not sufficient for these conditions of laser beam power, so when 2.0g/min was used the deep alloying zone started to be clearly visible.

The laser surface alloying at 2.0kW of laser beam power and high chromium powder feed rate of 4.5g/min (Fig. 10) result in full depth of alloying, the transient zone is very narrow (Fig. 11) and majority of alloying zone is chromium enriched. Formed microstructure is composed of cellular-dendritic crystals with chromium microsegregation at cell boundaries and massive ferritic grains with needle-like austenite (Figs. 11-12). At the top of alloyed surface, where none fully dissolved powder particles are embedded the small secondary porosity is present (Fig. 10).

Fig. 13 shows the microhardness depth profile of laser alloyed surface. The hardening effect of chromium alloying is visible on surface depth up to 0.6-0.8mm (for laser beam power of 2.1kW), microhardness in this zone reaches about 250 $HV_{0.05}$ and then gradually decreases to value characteristic for the substrate, ca. 100 $HV_{0.05}$. Higher microhardness at the top of alloyed surface (about 300 $HV_{0.05}$) is associated with pure chromium powder particles presence.

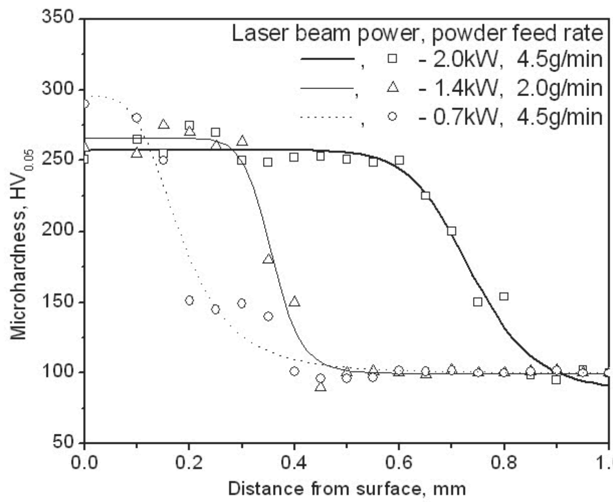

Fig. 13. Microhardness depth profile of sintered 316L after laser alloying with Cr at different laser conditions of laser beam power powder feed rate

The EDS microanalyses were carried out in the cross-section of laser surface alloyed stainless steel (Fig. 14). The concentration of major chemical elements undergoes variations along penetration depth of alloyed zone (Fig. 15). Concentration of Cr is higher on the surface top where embedded pure chromium powder particles can be present (Figs. 16, 17), therefore the concentration of 37%Cr was registered. Altogether with an increase of powder feed rate and laser beam power chromium enrichment of austenitic microstructure takes place and the formation of ferritic phase occurs. The initial quantity of chromium ca. 18-20% increased to 26-28% in the center of alloyed region (EDS analysis no. 2 in Table. 3). The fluctuation of alloying elements, more evident on the chromium level is due to the microsegregation at the solidified cell boundaries.

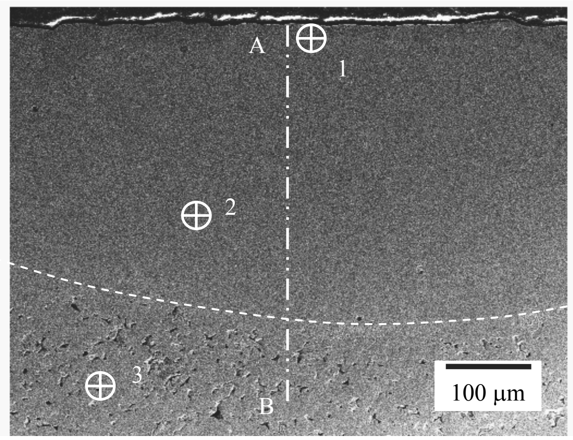

Fig. 14. The cross-section of laser surface alloyed 316L at 1.4kW of laser beam power and powder feed rate 2.0g/min. The dashed line represents the interface between remelted zone and base metal

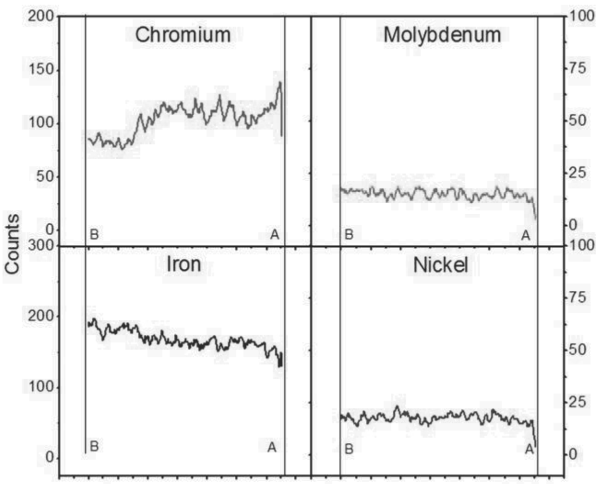

Fig. 15. The liner distribution of chemical composition along dash-dot line according to Fig. 14 on the cross section of Cr alloyed surface of 316L

Table 3.
The EDS point analysis according to Fig. 14 on the surface alloyed at 1.4kW of laser beam power and powder feed rate 2.0g/mi

Element	Point analysis, wt. %		
	1	2	3
Cr	37.65 ± 0.53	26.32 ± 0.46	20.07 ± 0.41
Ni	8.27 ± 0.42	7.91 ± 0.43	8.77 ± 0.44
Mo	2.26 ± 0.51	2.90 ± 1.68	3.45 ± 0.55
Fe	51.82 ± 0.60	62.87 ± 0.62	67.71 ± 0.64

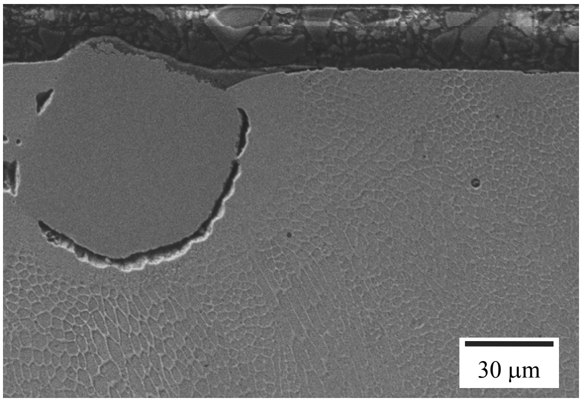

Fig. 16. The chromium powder particle embedded on the surface of 316L stainless steel alloyed at laser beam power of 1.4kW and powder feed rate of 1.0g/min

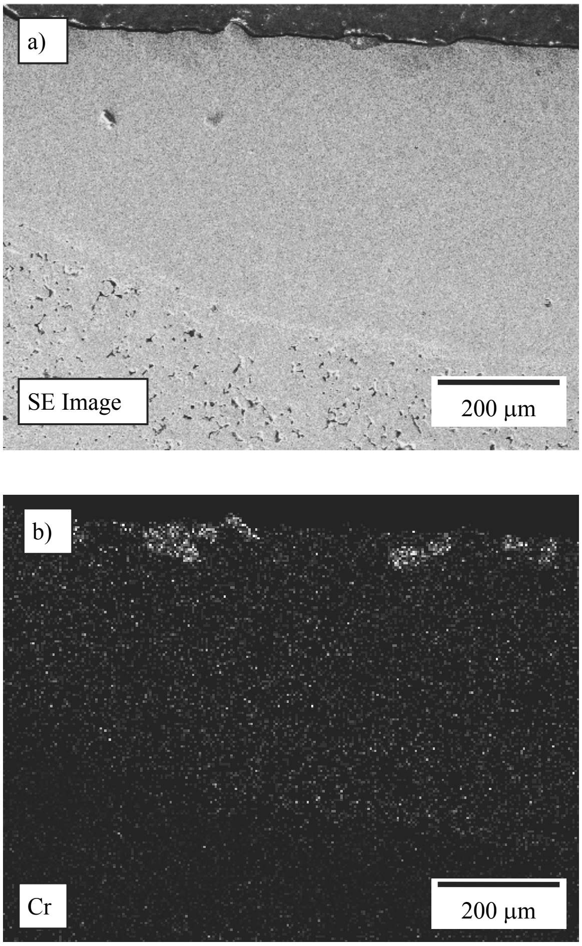

Fig. 17. The image of laser alloyed 316L at laser beam power of 1.4kW and powder feed rate of 2.0g/min, a) SE-image of cross section and b) the map of chromium distribution

4. Conclusions

Studies of the influence of laser alloying conditions, both laser beam power and powder feed rate on the microstructure and properties of chromium alloyed sintered austenitic stainless steels can be summarised as follows:

- Increase of laser beam power resulted in slight increase of alloyed surface width, while the penetration depth increased more remarkably from 0.5mm for laser beam power of 0.7kW to 0.85mm for 2.0kW. The surface of alloyed layer has a silver metallic shine and formed weld bead is uniform and flat without any undercuts or superficial cracks.
- The roughness of alloyed surface comparing to unalloyed one increased triply to Ra=4.7µm for powder feed rate of 4.5g/min at 2.0kW of laser beam power. The roughen surface of Ra>7µm was obtained at intermediate laser alloying conditions i.e. 2.0g/min of powder feed rate and 1.4kW of laser beam power. Taking into account obtained results, the additional surface finishing treatment is indispensable to ensure satisfactory surface roughness of alloyed surface.
- The microstructure of laser alloyed layer is composed of austenite and ferrite, as confirmed by x-ray analysis and can be called duplex microstructure. The chemical composition of alloyed stainless steel surface: 26-28%Cr, 8-10%Ni, 2.5-3%Mo corresponds to ferritic-austenitic region in the well known Scheafler diagram. The alloying process influenced the microstructure refinement and formation of cellular-dendritic crystals with the chromium microsegregation at the solidified cell boundaries. Such type of microstructure is present when alloying was done at lower laser beam power and lower powder feed rate. The fully alloyed chromium zone also consists of massive ferritic grains with the needle-like austenite precipitations on the grain boundaries. The initially formed ferritic phase of chromium enriched region undergoes a partial transformation to austenite and is characterized by presence of Widmanstatten austenite plates which nucleate and grow from the grain boundaries.
- The ferrite phase content after surface grinding treatment evaluated on the top of alloyed surface was varied in the range of 33-38%.
- The laser alloyed surface hardening effect is mainly caused by complete reduction of porosity as well as chromium solution hardening of refined microstructure. The microhardness of surface layer increased from 100 to $250HV_{0.05}$ on surface depth up to 0.6-0.8mm.

Obtained results are very promising in respect of possible application of HPDL laser in surface alloying for sintered stainless steels. Despite of lower thermal conductivity of sintered steel comparing to wrought one they are still easy to process by laser treatment. Produced alloyed layers are uniform and flat without any visible undercuts or superficial cracks. The high surface roughness requires the application of final surface finish treatment but obtained penetration depth is sufficient to perform it. Laser surface alloying with Cr of PM austenitic stainless steels provide the possibility of simultaneous porosity reduction and the formation of specific material properties in surface layer, particularly in respect of corrosion resistance that will be studied in further works.

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INNOVATIVE ECONOMY
NATIONAL COHESION STRATEGY
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