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MICROSTRUCTURE CASE STUDY OF LENS^{tm} PROCESSED CYLINDER FROM AISI H13 STEEL

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ABSTRACT

This paper presents the microstructure analysis of laser cladding process fabricated cylindrical specimens from AISI H13 steel, produced parallel to parts of tools for deep drawing, by LENS^{TM} 850-R, OPTOMEC, equipment. The specimens are with dimension Ø14,5x15 mm, made by 1000W laser power moved with 60 mm/s traverse velocity, formed on substrate from low carbon commercial steel.

The showed macro- and microstructure is typically to that presented in investigation from several authors, related to build parts by additive deposition of layers from molt bead created by laser and direct addition of powder in melt. The LENS formed structure of dense body is similar to welding with non equilibrium solidification of melt by fast cooling. The microstructure is non uniform in all direction in body, influenced from LENS process, as well as from non uniform temperature fields, cooling rate and different direction of heat transfer. Generally microstructure consists from dendrites and equiaxial grains built from melt or by overlapping of deposits influenced areas and heat influenced zone. The microstructure is formed of: (1) martenzite obtain with phase transformation from primary solidified austenite and (2) rest intercellular ferrite, rich of alloying elements Cr and Mo.

***Key words**: AISI H13, LENS process, microstructure, rapid cooling solidification, non uniform solidification.*

1. INTRODUCTION

Laser technique finds more and more application in manufacturing or machining parts, especially within the Solid freeform fabrication (SFF) process for tools and moulds. It is already developed and applied in the industry, more procedures of using the laser light as heating and melting energy for different material and metals for welding and smelting. By using CAD computers platform, it is possible together to blow powder and smelt the powder by movement of laser beam. It is developing processes for powder sintering by using low energy laser for porous bodies and strength energy laser for full dense bodies. Among the implicated processes are: Selective Laser sintering (SLS) for porous parts and SLS/HIP (High Isostatic Pressure) – combination of laser and conventionally sintering technology, processes for direct forming solid dense parts, for which very small mechanical finishing is necessary. At last, there are the techniques Selective laser Melting (SLM), Laser Engineering Net Shape (LENS) and Direct metal deposition (DMD) which have produced components with high performance [1,2].

Laser Engineered Net Shaping (LENS^{TM}) is a laser assisted, direct metal deposition manufacturing process which was developed by Sandia National Laboratories and was commercialized by Optomec Design Company in 1997 [3]. The LENS^{TM} process is an additive manufacturing process in which the parts are directly fabricated from a 3-D solid model line by line and then layer by layer. In the LENS^{TM} system, a laser beam is used to create a molten pool on the base or substrate on which the laser beam is focused. The powdered particles are then introduced into the molten pool. As the laser beam moves over the powdered metal, it melts the powdered metal and once the laser beam moves in the forward direction, the molten metal solidifies (Fig. 1). A layer is deposited by moving the substrate on the control table in the x and y directions. Once a layer is deposited, the next layers are deposited by incrementing the laser and the powder delivery nozzle in the positive vertical direction. This process continues until the whole part is completed [4]. The fabrication process takes place in an argon controlled chamber for preventing oxidation of the fabricated part as well as the powdered metal [5].

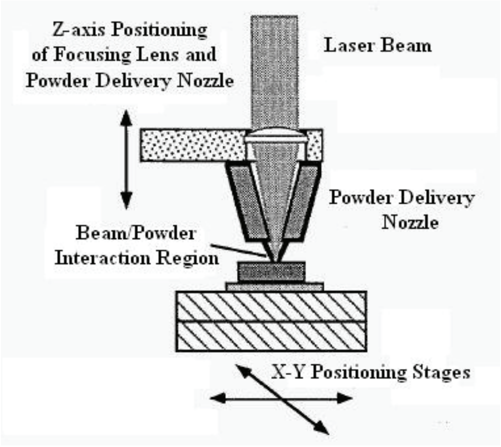


Figure 1 - Scheme of the LENS process.(Transmited from [7,8])

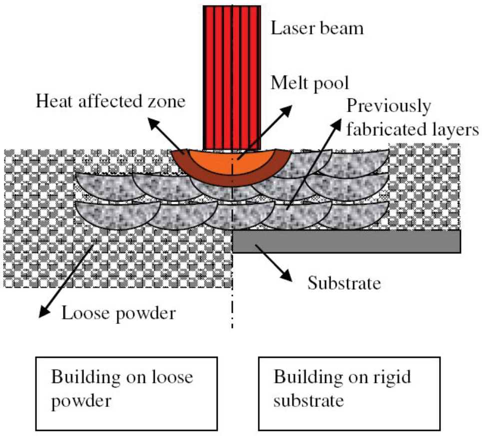


Figure 2 - Forming of layers by different passes of laser by building smelt pools in line (in vertically section). (Transmited from [9])

Influence of number of deposited layers (dimension of good) by free forming of goods from AISI H13 steel to formed microstructure, temperature fields by multiple passes of laser beam, are the subject of investigation of many interested parties [3÷8,11,12]. Properties and evaluation of microstructure of fabrication parts by LENS process is very complex due to the thermal changes of alloyed system and non equilibrium distribution of alloying elements to different phases [7]. Moreover, some general perceives can be illustrated by discussion of specifics on Cr and Mo alloyed Fe system and by model developed thereof [7], which goal is to describe the response of alloy to processing conditions (Fig. 2). Tool AISI H13 steel is a good example of alloying system which are developed and happed by building the microstructure during processing. LENS process on thus steel is already described in details in [8]. H13 is commercially accessible secondary thermal strengthened alloy with very high industrial importance, if the heat treatment is made by classical metallurgical production and additional heat treatment, by building carbides which content Mo and Cr.

Microstructure of LENS free formed goods. The formation of microstructure from H13 body build with LENS process was treated in different aspect. Very important aspect is to overlap the temperature change in built line, if one or more new lines or layers are added over it. Every new line and layer, re-melted part of previous pass, could cause the temperature changes in already solidified lines or layers. The temperature changes are influenced by heat transfer from the last melted phase to environment. The heat transfer must be different to different direction, depending on the temperature of the neighbouring area and its thermal conductivity coefficient.

Starting from over facts, the changes of temperature measured in a line, by addition new line and layer, are measure by [8]. The cited authors have related the built microstructure to the H13 phase diagram (Fig. 3). They have noted that non-equilibrium conditions exist given the rapid heating and cooling rates. The microstructure shown from the upper portion of a freeform body to bottom, and its view, can be separated into three different regions. Also, shown in the height of the individual built passes, could be remarks of three regions.

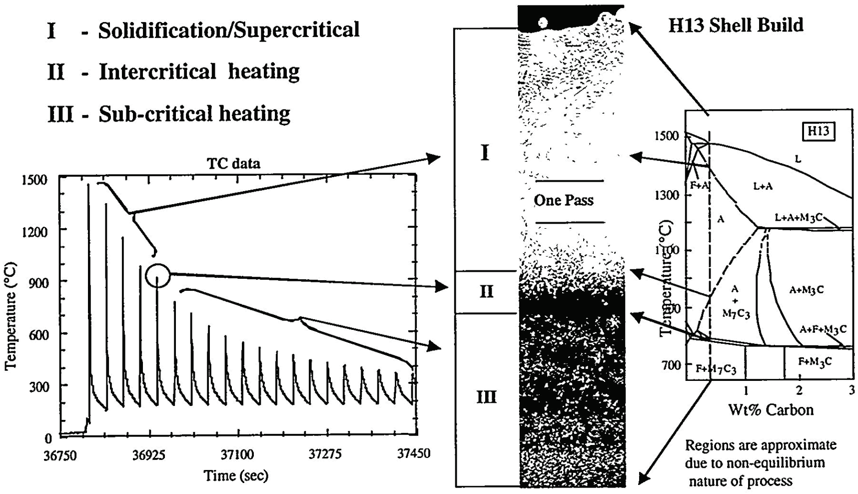


Figure 3 - The overlapping of measured temperature on the first layer with phase diagram of H13 with 0,4 mass% C where new 20 layers are added. For the description of properties are used three temperature regions. The high of laser pass is 0,25 mm [from [7].

Region I is composed of as-solidified H13 (last pass) and supercritical reheated material. It is noted that it is the last pass re-melted part of the previous pass. Thermal cycles corresponding to these regions are also shown in Figure 2. This supercritical cooled region has some segregation of alloying elements occurring as a result of partitioning during solidification, and little homogenization occurring due to the slow diffusion rates in austenite [10]. The exception is carbon for which the diffusivity is much more rapid and a uniform distribution is expected [10]. Microprobe measured segregation ratios (interdendrite columnar)/(dendrite cores) is: Cr = 1.3, Mo = 1.2, and V= 1.5. The supercritical region extends from the liquids temperature to the ferrite + carbide two-phase region, which on the equilibrium diagram is -925°C. The light etching material of the supercritical region is no tempered martenzite (hardness of 59 HRC) in which carbides are not detected using transmission electron microscopies (TEM [7].
Region II corresponds to material cycled into the intercritical two-phase and narrow three phase regions shown on the diagram. This material had been previously cycled into the supercritical region, and corresponds to the first dark etching region noted in Figure 2. It is interesting that the height of this region is close to that of a single pass height, and based on the phase diagram, would have contained a thermal gradient of 600°C/mm [8]. For the thermal cycles shown, only the peak temperature of the fifth thermal cycle lies within this region.
Region III contains the material that in addition to having experienced thermal cycles in the upper two regions, also experienced subcritical thermal cycles [8]. The final microstructure of last region consists of tempered martenzite with a bimodal area of V and Cr containing carbides [8].
Some properties of LENS AISI H13 freeform body and as heat treated are given in [13]. It shows influence of the body's high to microstructure and correspondent changes, the morphology of dendrites and carbides. With heat treatment the microstructures are changed, so the heat treated LENS structure and properties are close to conventional heat treated steel, with some benefits to toughness by LENS treated material.
Microstructure of each local section of specimen by laser welding of Cr content steel is different by dendrite and uniaxial grain scale, regardless of the velocity of laser moving or solidification rate [14, 15]. Investigated moving rate was low in comparison with kinetically limitation of growth, so the solidification must be followed by heat transfer which results at different grain scaling and crystal orientation [15]. The thermal gradient by LENS process, as noted, are directed by Maragoni convection of heat, which is resulting in unstable solidification process [14]. Additionally, it is noted that remolding process occurs on the previous deposited layer. Because the dendritic growth is not easily possible, the average distance between cells is different and can be used as measure for the approximation of dendritic growth. In case of H13 steel, average distance between dendrites is 5.7 μm, and at the upper part is 7.4 μm [15]. That phenomena is also influenced by the heat conductivity change of material more than the cooling rate. In H13 steel, heat conductivity changes have greater effect then the cooling's rate and already formed structure than the laser power and its passing velocity [15]. The morphology of the structure by laser welding is primary austenite and intercellular ferrite. Austenite is primary solidification phase and the rest is ferrite rich on solved Cr and Mo.
The purpose of this paper is to make practical contribution to estimation of microstructure by LENS process produced tool parts and to make useful metallographic examination by controlling the production and estimating properties of part. Along with cited authors, the accessible funds and knowledge from numerous consulted authors remarkably help the explanation of the microstructure.

2. EXPERIMENTS

To estimate the structure of tool for deep drawing, produced by freeform LENS process at foreseen quality of steel material AISI H13 (WN 1,2344) two parallel produced cylindrical specimens in same condition and together with the toll parts were used.

Product tools and specimens are formed from Fe powder and powdered alloying elements and carbides which corresponded to standard AISI H13 steel. Chemical analysis of expected and real estimated content of steel is to be shown in Table 1. The chemical analysis is made by "RZ Technical control" J.S.C. Skopje, R. Macedonia. The specimens are formed by TIC-LENCE d.o.o. Celje, Slovenia on the equipment LENS^{tm} 850-R, produced by OPTOMEC, USA [www.optomec.com].

Some characteristics of the used LENS equipment are: 5 axial moving of the plat on which are build parts and specimens, protection atmosphere – argon with 10 ppm O_2, 0,5 kg/h capacity of materials, used laser movement - 60 mm/s, power – 1 kW IPG Fiber Laser.

Table 1. Chemical content of AISI H13 (WN 1.2344, X40CrMoV51, Č.4753)

	C	Si	Mn	Cr	Mo	Ni	V	Rest Fe
Standard	0.38	1.00	0.38	5.15	1.35	-	1.00	-
Produced	0,34	0,95	0,31	5,30	1,23	0,12	1,02	-

Cylindrical specimens, with dimension Ø14,5 and H=15 mm are formed on support from wrought deformed low carbon commercial steel with chemical content: – 0,065%C, 0,04%Si, 0,25%Mn, 0,015%P, 0,018%S, 0,47%Cu, 0,019%Al, 0,21%Cr, 0,16%Ni, 0,001%V and 0,01%Mo. By cutting of cylindrical pediments, a part from support was also cut, which made it possible to investigate the content, microstructure and some of their properties.

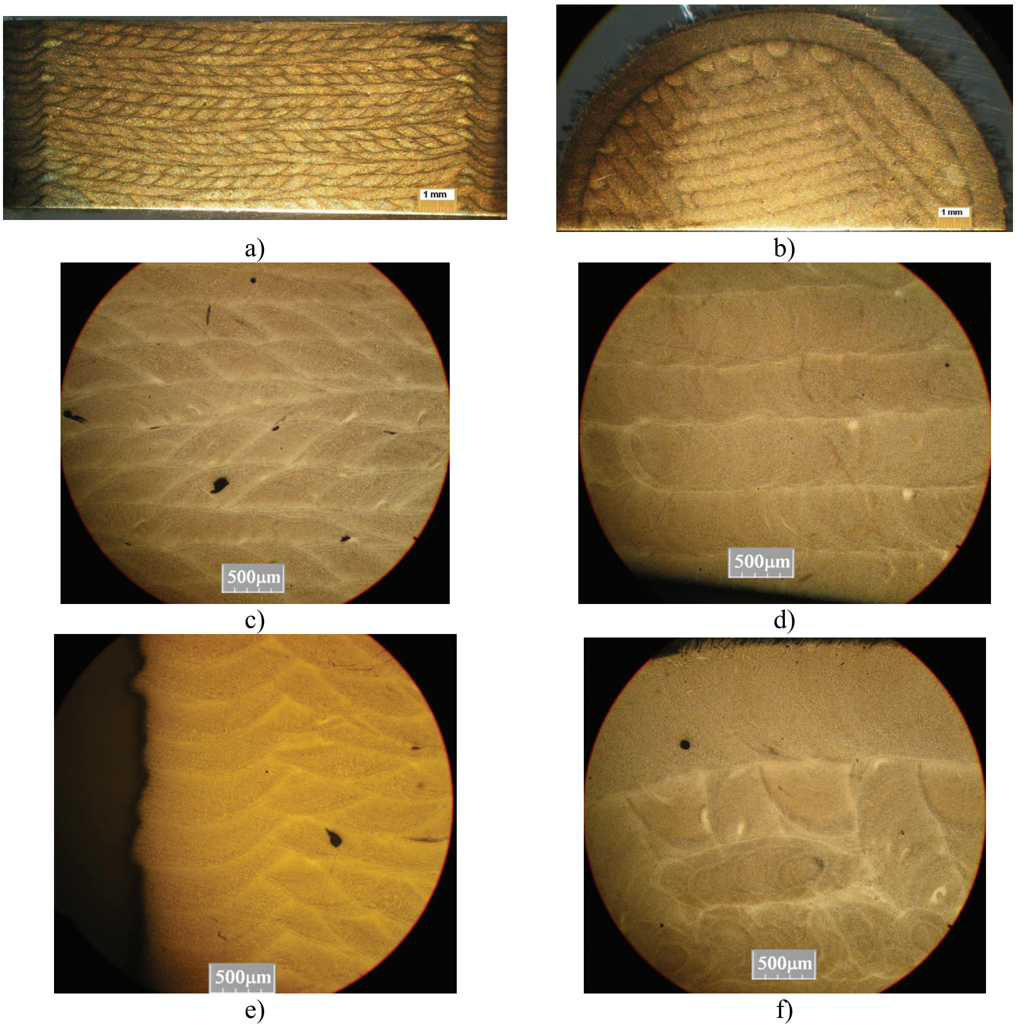
2.1 Macroscopic and microscopic investigation

The macrostructure was watched under macro digital camera. On the top circle area the surface structure is characterized by numerous protuberance with cylindrical or cone form on the circumference by side (see fig. 4) or line on top of cylinder.

Figure 4 - Structure of side surface of LENS produced specimen

One full cylindrical specimen is used for micro structural investigation on upper (last pass of LENS production) circle surface and microstructure of support and from the other one a small cylindrical plate is cut , in order to diameter cross-section and high-section for metallographic estimations. The cut were made by erosimat cut machinery. The full specimen is prepared for metallographic by optic microscope and the small parts cut by diameter and height was prepared by polyester.

Specimens for light microscopic examination are prepared by classical metallographic preparation – grinding with grind papers of SiC from the Struers – Denmark, on rotary universal equipment DPU-2, with SiC granulation 120, 220, 400, 800, 1200, 2400 and 4000, and additionally polishing with alumina paste with grain 5 μm on AP cloth.



a) b) c) d) e) f)

Figure 5 - Macrostructure of deposited lines of cylindrical specimen: a) cut by height b) by diametrical cutting (lighted in microscope by acute angle), c) micro photographed by increased magnify of lines and layers cutting of cylinder by height, d) photographed by increased magnify the lines and layers by diametrical cutting, e) microphotography near the wall end of cylinder of height cut area, f) microphotography near the wall end of cylinder of diametrical cut area.

The polished specimens were rinsed with ethyl alcohol. The polished cross section surface was observed by optic microscope Neophot2. Only a few number of process inherent pores are recognizable in the microstructure. Most of the pores are normally detected in the overlapping areas between the beads, but therefore the number of pores which is kept in the microstructure is relatively small. The material can be classified as very clean on inclusions, except of isolate carbide inclusions showed by some area and pores, which reach dimension cca 100μm (Fig. 5c, 5e, 5f).

Microstructure etching is made by 4% Nital (Nitric acid in Ethyl Alcohol) in duration to 10s to 30s. Longer time etching duration lead to possibility to see macrostructure on line of body's formation and the short time etching, make possible to see form of phase with microscope. The macro- and micro-structure were being watched by light microscope NEOPHOT 2, made by Karl Zeis - Jena.

Some example of macro and micro structure are presented in Figure 5 to Figure 11, with some remarks over showed structure given in figures.

The investigation also shows a change of the microstructure along the height of the part. This result is due to the changing heat condition during the deposition. As with any rapid solidification or welding process, the microstructure in any local section of the sample contains a variation in length scales.

Hardness on top of cylinder is measured by Rockwell's method and shows 54 to 54,5 HRC.

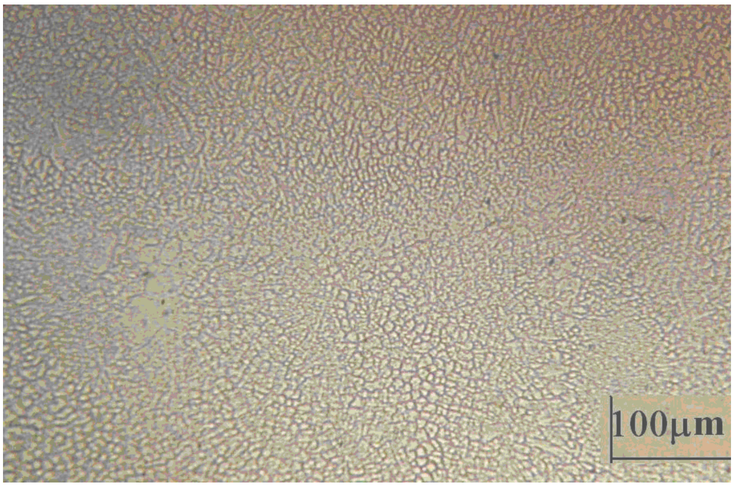
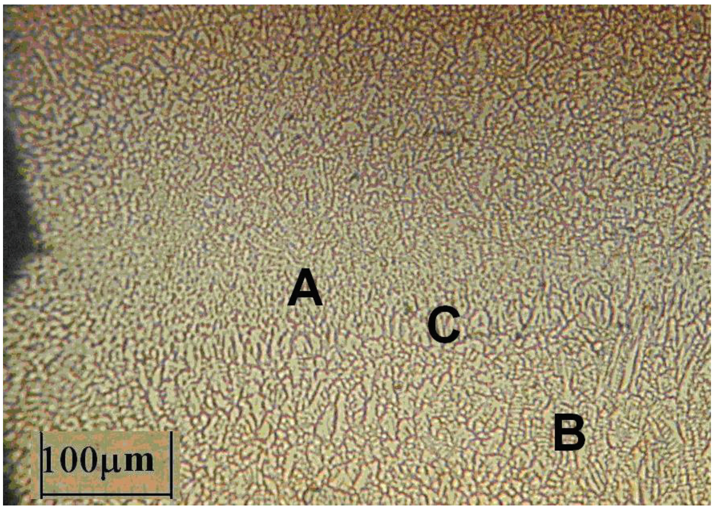


Figure 6 - Microstructure of the top polished surface from cylindrical specimen made by LENS process. Part of area with overlapping of two parallel lines on top layer. Slowly very small equiaxial grain or normal cut uniaxial grains, bigger grain from heat influence of last line pass and uniaxial grain formed by low temperature reheating.



7 - Microstructure of two parallel passes cutting of cylinder by height, near a cylindrical side. Slowly different grain form of grain: very small equiaxial (A), heat growth (C) and solidified from melt dendrites and uniaxial grain. (B). Overlapping of line passes cause the side un-roughness.

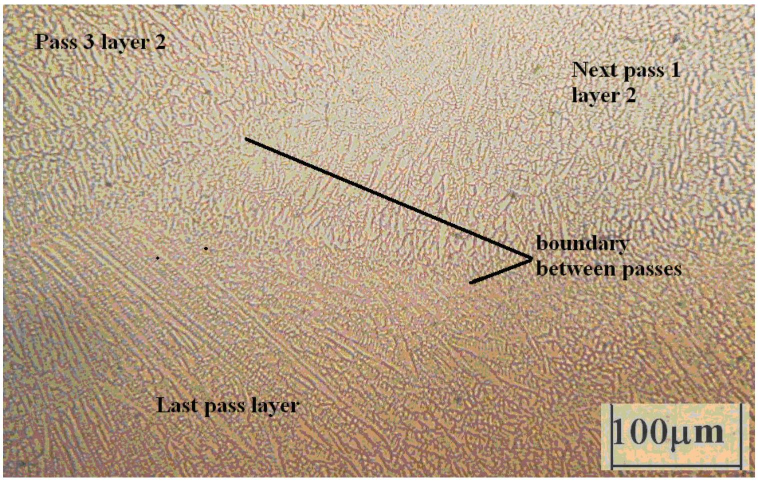


Figure 8 - Microstructure of normal section of deposits lines by forming the cylinder specimen, near the center of diameter. Visible tree different passes by forming of cylinder, bottom layer and two next passes. On the boundary between passes visible influence to structure from overlap's deposited lines. Large primary dendrites and uniaxial crystals of martenzite, late and cellular grain in followed pass (right top), with rest ferrite in between dendrites and intercellular areas.

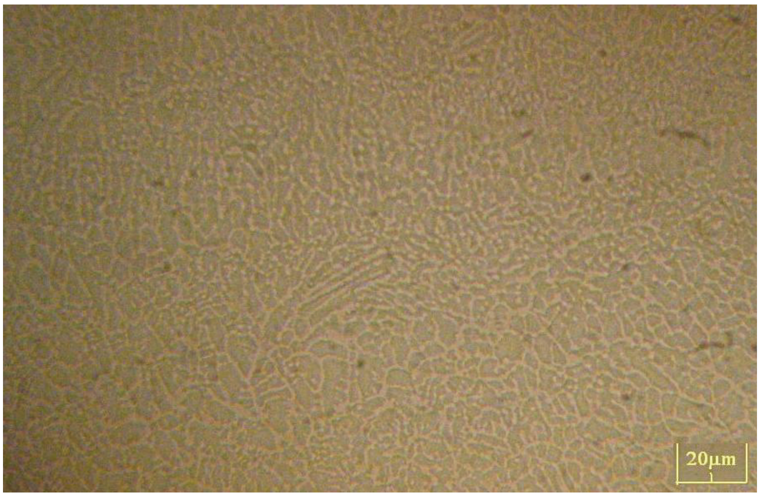


Figure 9 - Typical microstructure from diameter cut surface of cylinder. Visible martenzite equiaxial, some uniaxial maretnzite grain and ferrite in between. Structure is formed from heat influence of next layer deposits pass.

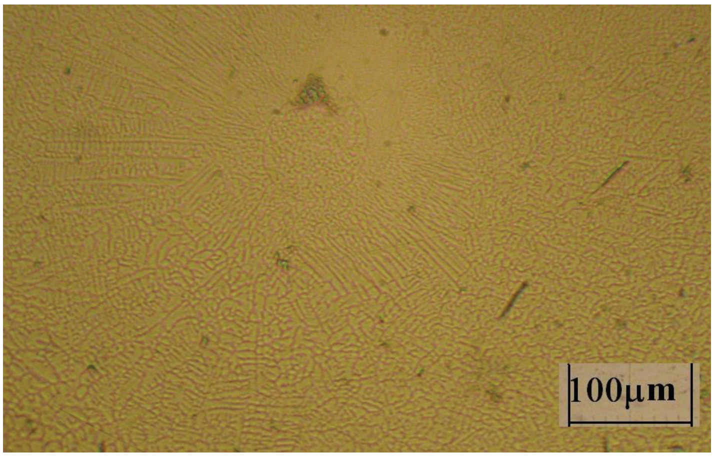


Figure 10 - Characteristic micrograph from top polished surface of cylindrical specimen formed at last pass by LENS processing. Visible crystals growth from a central melted area. At center equiaxial grain, follow by dendrits and end uniaxial grains, from primary austenite and transformed to martenzite and rest ferrite intercellular. (The needle forms are not inclusion – unclean photo lens)

3. DISCUSSION

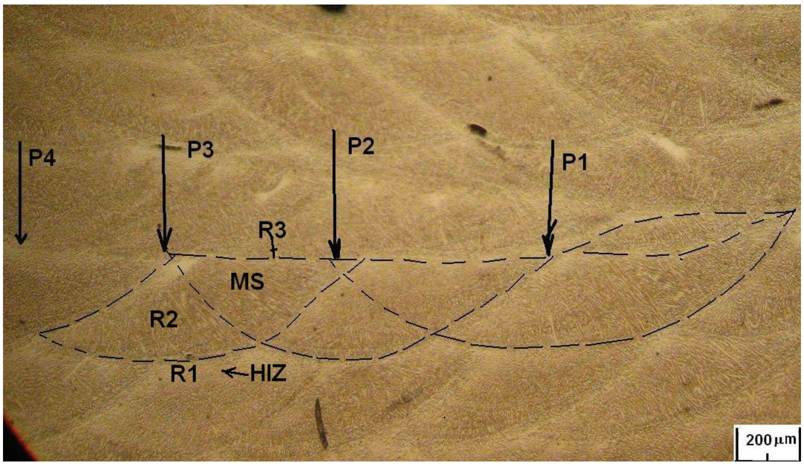
The macro structure of cylinder built by LENS process is typical for process which deposition of material is made by line addition. As is pointed in the introduction, by LENS process laser beam smelt the support or previously formed layer from base material with addition of powdered base material in smelt pool. Because of that, to avoid formation of porous solid body, the added layers shell be formed very close to each other, so that the layer overlapping previous line of solid material from last line and lines from last formed layer. Thus to realize, by formation of cylindrical body, lines with different large shell be added to form circle plate from more overlapped lines. Moreover, the formed cylindrical body is formed by different way of addition of melt pool, with interrupt of line smelt pool and single laser beam pool. Thus it is possible to show in the macrostructure visible in cut surface in height and diameter of cylindrical specimen in light optical structural examination, and give the results from experiment.

The macro structure built of structural elements is different in both direction of body growth – vertical and transversal section of specimen, which is practically visible in Figure 5a to 5f, by different magnification. In the circle section tone can see that on the diameter a continuity line is formed, in deferent to inner line of smelt pool lines, which have different direction and dimension. Figure 5a to 5f display cross- and vertical sections of the laser formed H13 steel cylinder. It is to note that when the laser moves away, the melted layer begins to solidify. The cooling rate in the liquid state is very high as a result of self-cooling, and Cr becomes enriched in the remaining liquid regions and dendritic structure forms. As noted in [14], the basic microstructure of the dendrites could be surrounded by an extremely fine lamellar structure that was constituted of metallic carbides, martenzite, and, probably, retained austenite caused by the mono-variant eutectic reaction, which is impossible to see by optical examination.

The vertical section of specimen show the built from horizontal layers, but with different way of their formation, some from discontinue, with very approximate form of rhombs and others from continue layers. The last layer, which builds the diameter of circle, has different interrupted form. Thus evidently gives explanation on formation of diameter by transversal section.

By higher magnification, the microstructure of different, already described parts of macrostructure, shows as different areas by vertical section of specimen, seen as areas composed of separate crystal or crystal grains with different habits: polygonal (equiaxial) alotriomorphic (epitaxial), dendritically and lathery grains (uniaxial) (see Figure 5 to Figure 10). This structure indicates that it is formed by solidification of melt and phase transformation during cooling, corresponding to equilibrium phase diagram or non equilibrium for the H13 system. Direction of grains growth note to heat transfer. Different grains form in a region indicated to different heat transfer in a region, which is, with some exception, repeatable by next region. Epitaxial grains end with fast braking by next region, with built equiaxial quite disperse grains (Figure 8). This has shows to undoubtedly influence the next pass and remolding process on already solidified region in the same manner (Fig. 11). However, it must be noted that that equiaxial and round form grains are actually mostly cut lathes and epitaxial grains. As noted in [7, 8] there are three remarkable regions of microstructure forming, corresponding to structure formed by solidification, tempered grains growth by high temperature range and phase transformation by fast cooling. Generally, all this grains form are from martenzite, built from primary solidified austenite, by fast cooling and tempering, with intercellular formed ferrite, rich on alloying element, which are not in state to bring equilibrium distribution between austenite and liquid phase by over melt temperature [14].

Interesting case of structure is noted at layer close to top cylinder surface, shown in Figure 10. From a sphere forming center, those have equiaxial very fine grains, to different direction growth dendrites, passing after some distance to globular grains. All that point out that the sphere region is very fast cooled and around from melt solidified the grains, which form is directory by heat transfer and by composition under cooling. That pointed to influence of already solidified pass or layer to next added layer by part's forming. All this processes could be having influence on properties of free formed parts, especially to retain tension in partial body region.



By height cutting show surface

P – pass; MS – Solidification from melt; HIZ – Heat Influenced Zone; R1 - solid stat re-crystallization region; R2 – Region of solidification from melt (from bottom layer); R3 – Region of small equiaxial grain (from new formed layer)

Figure 11 - Micro picture and explanation that have slow influence of next passes by form a cylinder by LENS process on the microstructure of different structural areas.

Formed macro and microstructure by LENS freeform parts from AISI H13 steel, on the basis of carried out examination and microstructure analysis, noted that they are built at very different and changeable temperature and concentration under cooling conditions, which all contribute to very changeable temperature and concentration fields, different diffusion on alloying elements and their distribution on different phase. Generally, the structure is formed by solidification from melt, with additional phase transformation, influenced by reheating, remolding and annealing processes. Formed structure noted that models of influence of cooling rate and tempering from next passes and build new melt pool of structure forming in [7,8] is a good basis for understanding the micro structure formation by LENS processes. The analyzed microstructure in this case study is corresponding to LENS process condition and phase related to no equilibrium phase diagram of AISI H13 steel. The obtained microstructure is far more different relatively to commonly wrought or sintered steel structure, where the structure of tempered martenzite and carbides of alloying elements, by commonly heat treatment dominate.

Different built structure by layers provoke the residual shear stresses between different layers by LENS process' fabrication of parts [12], what shall bring to different properties across height of parts, what is manifested, for example, at different hardness on bottom and top of parts and possible forming of cracks. In some area on outer diameter of cylindrical good some transversal to high crakes are remarked. The top region of parts could have higher hardness because of influence of tempering condition in the lower region. The obtained hardness of top surface from more then

54 HRC confirms the discussion to formation of martenzite in LENS formed structure and is higher as common heat processed H13 with maximum 54 HRC by dispersive tempering of quenched martenzite.

4. CONCLUSION

The microstructure and measured hardness of two specimens in cylindrical form from AISI H13 steel, produced by LENS process in the some condition with parts of tool for deep drawing, on support plate from common low carbon wrought steel were examined by light microscope. Hardness on top of cylinder is 54-54,5 HRC, which corresponds to martenzite formed microstructure. Macrostructure of cylinders demonstrates that it is formed by different diametrically layers build with more passes of laser. Formed macrostructure is different on cross and vertical section on cylinders. Microstructure shows that it is formed of different grains in scaling and morphology – mostly dendrites, lathes and globular grains, with very high probability from martenzite, built during primary solidification of austenite and follow phase transformation to martenzite and tempered, correspondent to chemical composition, carbides, as rest interdendritic and intercellular ferrite enriched with alloying element, dominantly with Cr and Mo. The microstructure of cross section of a deposited line of material shows that it is formed by solidification from melt pool, formed of laser beam, with overlapping on next pass with the previous, which causes the remolding of earlier passes an under layer and reheating part of dendrites and other arts of grains to their structural changes and formation of tempered structural zone (Heat Influenced Zone).

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PROUČAVANJE MIKROSTRUKTURE OBRAĐENOG CILINDRA OD AISI H14 ČELIKA POMOĆU LENS^{TM} SISTEMA

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REZIME

U ovom radu analizirana je mikrosturktura cilindričnih uzorka od čelika AISI H13 dobijenih spajanjem pomoću lasera na LENS^{TM} 850-R, OPTOMEC, uređaju. Pri izradi uzoraka dimenzija Ø14,5x15mm, primenjen je laser jačine 1000W i brzine 60mm/s sa osnovom od niskougljeničnog komercijalnog čelik

Prikazana mikro i makro struktura javlja se i u istraživanjima drugih autora, kod delova proizvedenih deponovanjem slojeva rastopljenog materijala, a koji je dobijen pomoću lasera uz direktno dodavanje praha u rastop. Struktura obrazovana pomoću LENS-a je slična kao kod zavarivanja sa neravnomernim očvršćavanjem rastopa pri brzom hlađenju. Ova mikrostruktura nije homogena u svim pravcima, što je posledica kako samog LENS postupka, tako i različitih temperaturnih polja, brzine hlađenja i smera prenosa toplote. Mikrostruktura se sastoji od dendrita i aksijalno-simetričnih zrna formiranih iz rastopa ili preklapanjem zona sa izraženim uticajem deponovanog materijala i toplote. Mikrosturktura je nastala od: (1) martenzita dobijenog iz fazne transformacije primarno očvrslog austenita i (2) ostatka interćeliskog ferita koji sadrži dosta legirajućih elemenata (Cr i Mo).

Ključne reči: *AISI H13, LENS proces, mikrostruktura, brzo očvršćavanje, nehomogeno očvršćavanje*