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Microstructure and Properties of Laser Additive Deposited of Nickel Base Super Alloy Inconel 625

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Abstract

Article presents results of laser overlaying welding of metal powder Inconel 625. Laser metal deposition by laser engineered net shaping (LENS) is modern manufacturing process for low scale production series. High alloy materials such as Inconel 625 nickel based super alloy have high thermal resistant and good mechanical properties, nevertheless it's hard to machining. Plastic forming of high alloy materials such as Inconel 625 are difficult. Due to high strength characteristic performing components made from Inconel alloy are complex, selective melting of metallic powder using laser beam are alternative method for Inconel tooling. Paper present research of additive deposition of spatial structure made from Inconel 625 metallic powder with CO_2 laser and integrated powder feeder. Microstructure analysis as well as strength characteristic in normal condition and at elevated temperature was performed. Possibility of using LENS technology for manufacturing components dedicated for work in high temperature conditions are presented.

Keywords: Laser additive manufacturing, Metallic Inconel625 powder prototyping, Additional material, Metallographic structure, Laser metal deposition

1. Introduction

Inconel alloy have wide range of application, due to thermal resistance in high temperature and mechanical properties research in using alloy 625 for industrial applications are widely studied. Therefore developing components from material with strength characteristic equal to Inconel alloy may significantly expand operating time. However manufacturing complex component made from nickel based super alloys are problematic, nevertheless using advanced prototyping technology for selective melting of metallic powder and accurate deposition process advanced elements working in high temperatures can be build. Moreover using LENS technology not only small, but considerably large elements (hard or even impossible to made using alternative technology) can be obtained. Components manufactured by laser deposition have rather good dimensional accuracy and surface quality, therefore for some applications are acceptable and after-machining processes are limited to minimum.

Additive manufacturing using laser beam for selective deposition of metallic powder combine with finishing process can be used as alternative technology for developing machine components. Due to obtaining high power density of focused laser beam deposition of Inconel can be easily performed. Laser melting of metallic powder are fully controllable process, energy density combine with coaxial powder delivery system allow to depositing molten powders [1,2,3,4]. Quick melting and crystallization process affect on metallographic structure of manufactured spatial elements. Thermo physical material properties change during deposition process, absorbed heat energy affect on beam absorption and crystallization rate. Metallographic structure change over deposition pass, liquid metal of additive and substrate material mixing, chemical composition of obtained overlay weld are combination of this two materials. In laser additive manufacturing relevant is that chemical composition of deposited material should be equal to substrate material, alloying elements could affect on crystallization process decreasing mechanical properties and caused defects. Properties of deposited material are related to chemical composition of material and deposition parameters. In laser deposition output power of laser and speed rate of process are essential. Speed rate of operation head are related to additional material deposition velocity, and affect on quality and quantity of deposition process. Therefore correlating of this two value are essential to rate of production. According to many studies when additional material deposition velocity is equal to process head speed ratio high shape quality factor can be obtained. Material obtained using deposition process, due to number of thermal cycles have different properties than base material, therefore properties and microstructure of manufactured material need to be tested. According to Inconel 625 properties and application possibilities many research of cladding and manufacturing of nickel based alloy is conducted, where microstructure, mechanical properties and corrosion resistance was investigated [5,6,7,8]. The main purpose of presented research is to argue, that LENS method using high power CO_2 laser and Inconel 625 metallic powder for obtaining spatial machine elements dedicated for components working in high temperature can be used. Due to prove this thesis tensile tests and fracture analysis of obtained material in normal condition, and at elevated temperature equal to 700^{o}C was performed. Moreover phase and structure transformation with hardness deposition and microstructure of deposited Inconel alloy 625 was studied.

2. Laser deposition of Inconel 625

Deposition of Inconel 625 was performed with Trumpf laser machine TrulaserCell 1005 provided with CO_2 gas laser TruFlow 6000 integrated with coaxial powder feeder GTV M-PF 2/2. Material used for deposition was metallic powder in grade Inconel 625, with particle size equal to -90+45µm and chemical composition (table 1).

Table 1.
Chemical composition of Inconel 625

Inconel 625	Cr_{max}	Mn_{max}	Mo_{max}	Fe_{max}	Nb_{max}	Ni_{max}
Substrate	20-23	0.5	8.0-10	5.0	3.2-4	58.0
Powder	21.8	0.4	9.1	1.6	3.66	62.7

Powder delivery are performed by inert transport gas (helium with flow rate 5l/min) and rotation disc dispenser with speed equal to 5rmp. Dispenser rotation speed and gas flow give powder feed rate equal to approximately 15g/min. To provide shielding atmosphere argon with flow rate equal to 15l/min was used. Substrate material in grade Inconel 625 sheet with thickness equal to 4mm and chemical composition as shown in Table 1 was used. Manufacturing process was performed using multilayer, alternately, parallel and perpendicularly deposition by laser engineered net shaping technology. Overlaying weld bead in each layers are deposited in same directions with interval equal to 2.5mm. Laser process parameters were optimized and with laser output power equal to 2.25kW, process head speed ratio equal to 0.8m/min cuboidal sample with dimension of 16x16x60mm was deposited (Fig. 1) [9].

Fig. 1. Deposition process of Inconel 625 metallic powder

Manufactured sample was machined and cylindrical sample for tensile and metallographic analysis was carried out.

3. Hardness test of deposited material

Properties of manufactured material can be determine using hardness test. According to PN-EN ISO 6507-1 standard hardness distribution in cross-section of deposited material with Innovatest Nexus 4303 was performed. Hardness test was performed via Vickers method with 10 kgF specimen.

To determine uniform properties of deposited material hardness distribution are carried out. Distribution of measure points are selected across sample axis and presented in the figure 2, therefore hardness of upper, middle and deposit to substrate zones are measured.

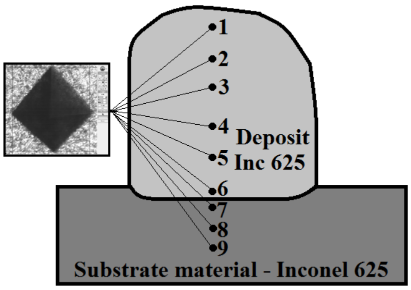


Fig. 2. Hardness point distribution of deposited Inconel alloy

Hardness distribution in cross-section according to test results was presented in graph form in the figure 3.

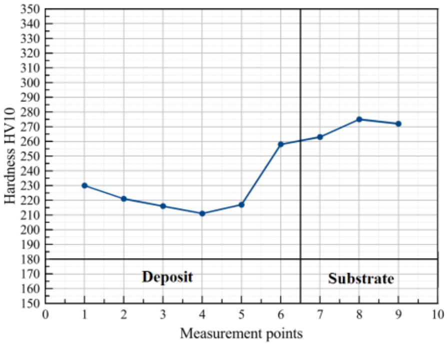


Fig. 3. Hardness of deposited material test results

Value of obtained hardness are between 210-277HV10. Hardness in upper layers are lower (maximum measured value equal to 230HV10, in deposit to substrate zone hardness are greater and take value from 258 to 277 HV10. Obtained hardness numbers are related to partial recrystallization process, material conductivity, heat capacity and diffusivity affect on change of metallographic structure. Hardness value in fusion zone and substrate material are approximately uniform. Heat accumulated in upper layers during deposition process affect on annealing process. Substrate and additional material chemical composition are similar, and material mixture do not affect on properties of deposited material [10,11]. No hardening process occur.

4. Tensile strength and fracture characteristic of deposited material

Metallographic structure of obtained deposited material is uniform, nevertheless some differences between upper and bottom fusion zone in grain size are observed. Fusion zone of additional and base material are narrow, not exceed 1mm. Equivalent of nickel, chromium, molybdenum and others components combining with thermal cycles during multilayer deposition affect on mechanical properties of deposited material. Melting and crystallization process changes the crystalline structure affecting on strength characteristics. In order to define properties of deposited material tensile test according to PN-EN ISO 4136 with Instron 4500 machine was carried out. Test was performed for normal conditions and at elevated temperature by using triple-fired channel type induction furnace integrated with testing machine. Sample was heated at the temperature of 700^{o}C and endure. Heating cycle is lasted 10 minutes in order to uniform temperature in the material volume. To confirm material properties, additional test was performed. Tensile test shown differences in strength characteristic (Fig. 4). Material stretched in normal condition have tensile strength equal to 690 and 686MPa, and at elevated temperature 610 and 603MPa, nevertheless plasticity of material are significantly differ. True strain of stretched materials in normal condition not exceed 4.5%, however in elevated temperature are equal to 10%. Endure material in elevated temperature affected in thermal stress reduction and material plasticity improvement [12,13].

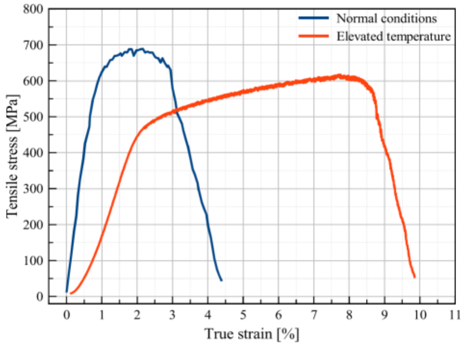


Fig. 4. Deposited Inconel 625 tensile test result.

Stress-strain curve shown increasing of material plasticity over tensile strength for material tested at elevated temperature equal to 700^{o}C. Comparing to base material, where tensile strength is in range from 724 to 896MPa, deposited material have lower strength characteristics. Moreover materials stretched in normal condition and at elevated temperature shown differences. Fracture in stretched material for normal condition are brittle (Fig. 5a), nevertheless test performed at elevated temperature shown transformation from brittle to ductile fracture (Fig. 5b).

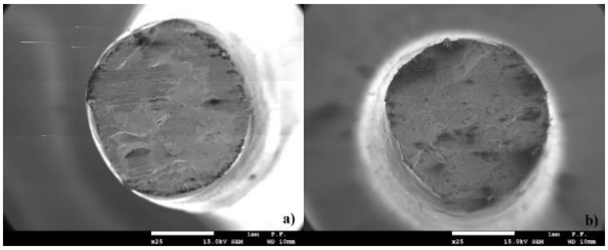


Fig. 5. Fracture test results of deposited Inconel 625: a) in normal condition, b) at elevated temperature

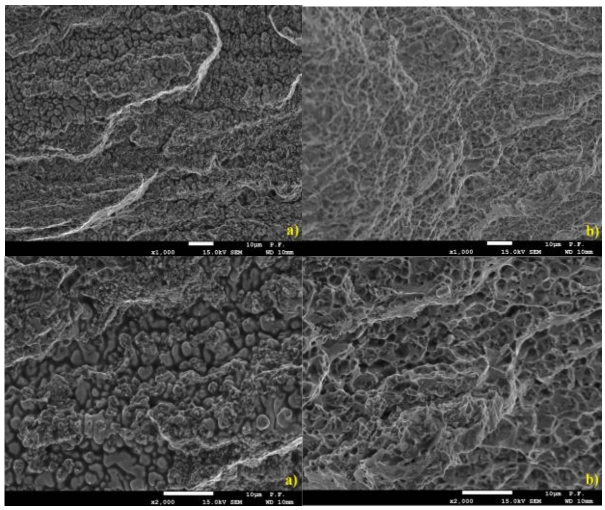


Fig. 6. Microstructure of fracture: a) in normal conditions, b) at elevated temperature

Fracture analysis shown dissimilarity in properties of materials tested in different temperature conditions. Tensile test performed in normal condition shown brittle fracture, sample analysis (Fig. 6a) shown granular structure and explicit crack boundaries with river pattern. Due to heat treatment microstructure are more uniform and shown elongated grains and predominance of transgranular cracking with parabolic shaped tear dimples. Fracture surface of the tensile test shown slip lines inside the grains (Fig. 6b). The slip bands may be formed during the plastic strain when strain-hardening occurs and may be responsible for a lower ductility. Material endure at 700^{o}C have equiaxial smaller grains, which can be related to recrystallization dynamics [14,15,16,17].

5. Microstructure of deposited material

Tensile strength test and fracture analysis shown dissimilarity in material properties at elevated temperature of 700^{o}C comparing to normal conditions. Therefore study of laser deposited Inconel 625 alloy (Fig. 7) crystallographic structure was performed.

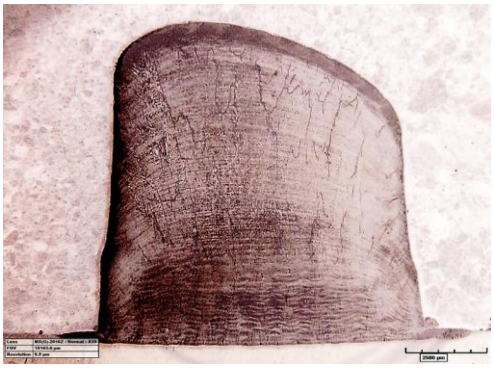


Fig. 7. Macrostructure of deposited Inconel 625 in cross section

Macrostructure analysis shown uniform material mixture with no porosity detection, cracking or bonding errors in deposited material. Transversal section revealed the pattern of layer-by-layer deposition. Therefore microstructure are characterised as columnar dendritc, oriented epitaxially from the substrate (Fig. 8,9,10). Moreover dendrites forming are related to direction of deposition process.

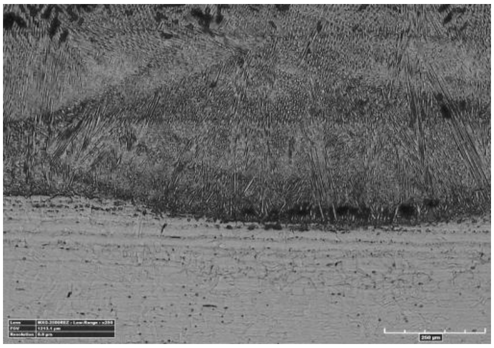


Fig. 8. Fusion zone of deposited and substrate material

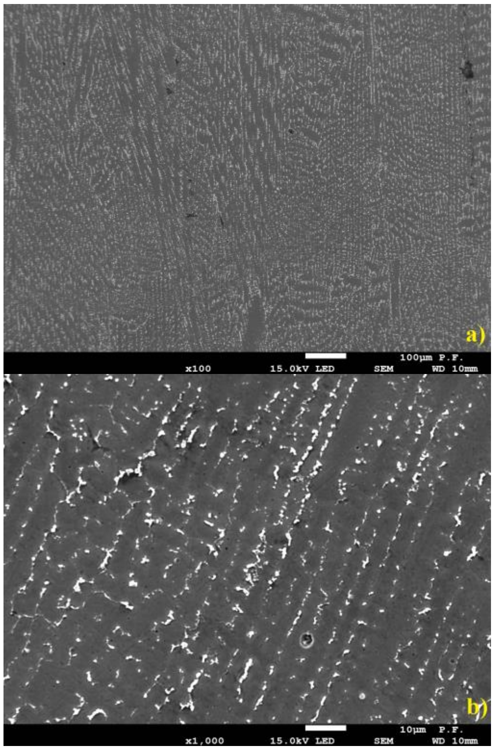


Fig. 9. SEM analysis of middle layers:
a) magnification x100, b) magnification x1000

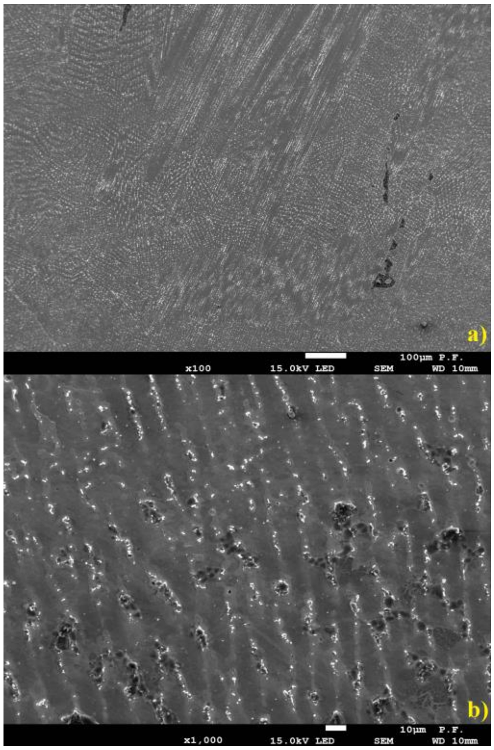


Fig. 10. SEM analysis of upper layers:
a) magnification x100, b) magnification x1000

Growth direction of columnar dendrites changed in each layer. When laser scan direction changes, the growth angle of primary dendrites changes to 45 degree with the substrate. In laser deposition, the substrate and deposited material affects in a heat sink. During solidification of molten material, cooling occurs via substrate and previously deposited layer. This effect determine directional growth of grains related to heat flux direction and subsequently formation of the columnar grains. The columnar dendritic growth epitaxial to primary dendrite from the partially remelted grains of the previously deposited layer. Temperature of the deposit at the back of the melt pool is relative high compared to the substrate and heat flux direction will decide of dendrites growth angle orientation. The cooling direction of the melt pool changes with the direction of laser scan. As a result, the heat flux direction are similar to the secondary dendrites of the previously deposited layer, however secondary dendrites are almost perpendicular to the primary dendrites. Therefore, during laser deposition secondary dendrites growth site for the primary dendrites of the top layer.

Microstructure of the transverse section of the each layer have two regions. Bottom part have a columnar structure and top fine dendritic structure with classical secondary dendrites. Due to the high solidification velocity of the bottom, secondary dendrites could not grow. As a result bottom part consisted of mainly primary dendrites [18, 19, 20, 21].

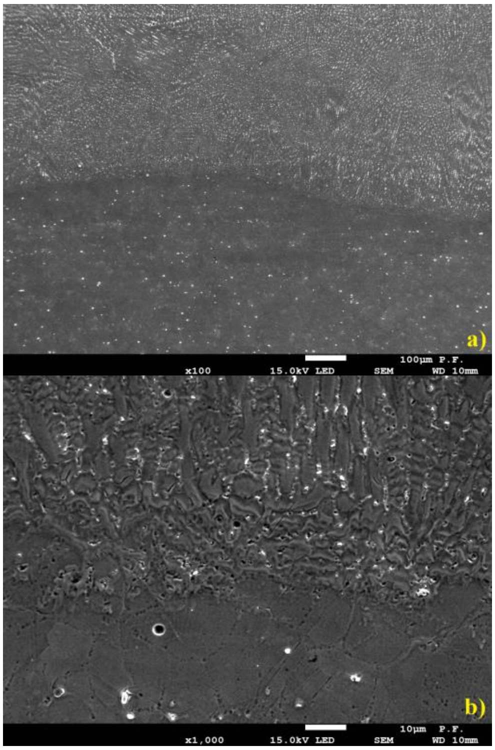


Fig. 11. SEM analysis of deposit to substrate fusion zone: a) magnification x100, b) magnification x1000

Laser deposition process are complex phenomena. Fusion between deposited and substrate materials may affect on elements migration, porosity and oxidation. To confirm uniform structure quality and quantity energy-dispersive X-ray spectroscopy analysis with scanning electron microscope JSM-7100F was performed (Fig. 12-16). Selected chemical elements distribution was shown according to defined measured line (Fig. 12).

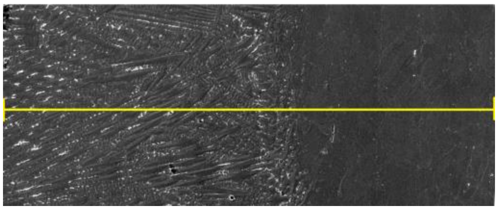


Fig. 12. EDS analysis line of deposit to substrate fusion zone

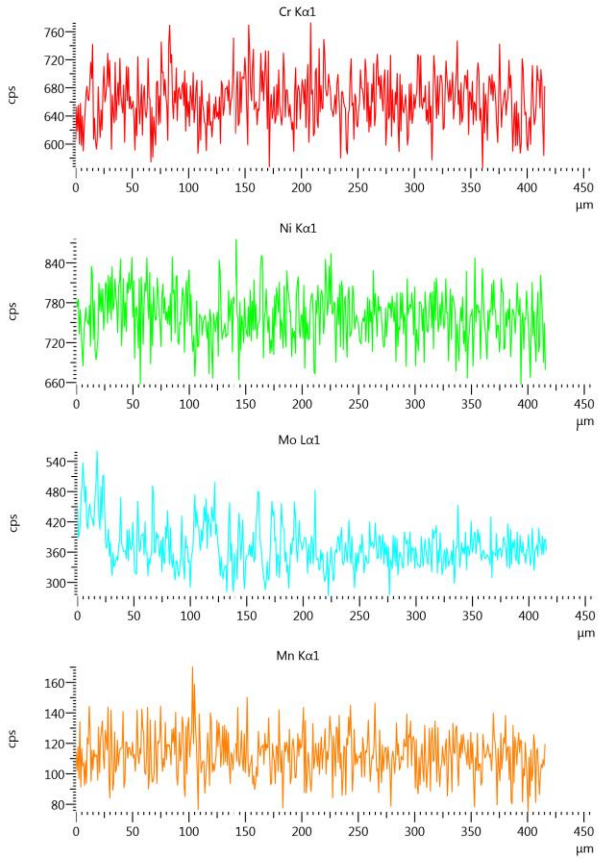


Fig. 13. Chromium, nickel, molybdenum and manganese amount alongside deposit to substrate measurement line

Chemical composition analyzed area shown uniform character, where no differences, high mixture ratio and linear chemical composition of deposit to substrate measured area.

Crystallographic analysis of interlayer fusion zone was performed, and area suspected as a micro-cracking (Fig. 10) was taken into further investigation (Fig. 14).

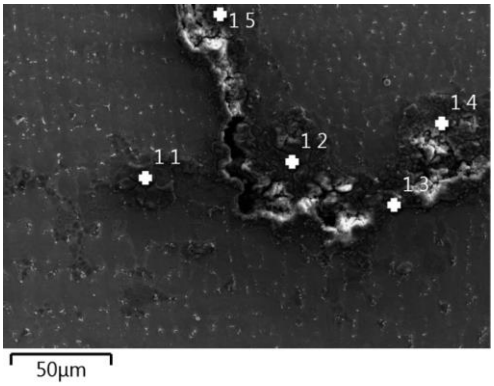
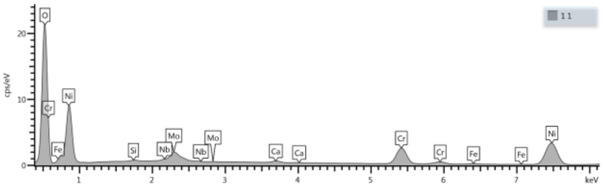


Fig. 14. EDS point analysis of interlayer inclusions

Performed EDS analysis shown in deposited zones precipitation in form of oxide separation (Fig. 14, 15). Therefore further studies was performed.



Spectr.	O	Si	Cr	Fe	Ni	Mo	Total
12	42.41	0.17	10.01	0.51	41.73	3.62	100.00
14	58.26	0.10	2.09		37.96	0.57	100.00

Fig. 15. Quantity analysis of interlayer precipitation

To confirm precipitation in form of oxides and exclude cracking appearance quality analysis of interlayer inclusion was performed (Fig. 16).

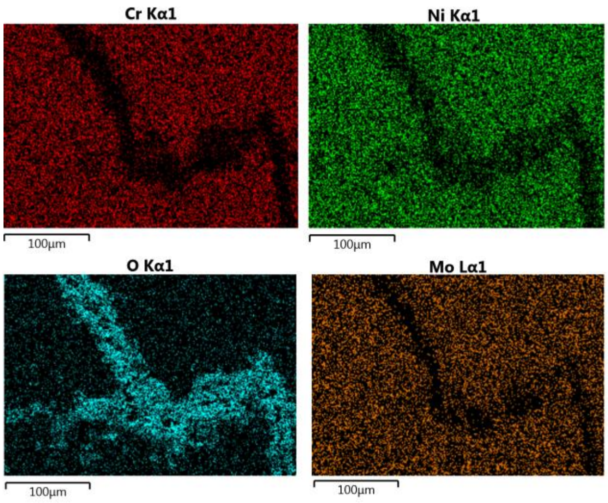


Fig. 16. Quality analysis of interlayer precipitation

Performed analysis shown oxides inclusion in interlayer zone, uniform map distribution of main deposited material elements and limitation in interlayer zone (except oxide) shown typical oxide inclusion assumed as nickel oxide.

4. Conclusions

Deposition process of Inconel alloy 625 using laser beam as a heat source was presented. Hardness distribution shown dissimilarity in material, value from 210 to 277HV10 was measured. Therefore lowest hardness number is in upper layers, and highest value in deposit to substrate zone. Annealing of upper layers can be related to heat accumulation in material during prototyping process. Strength characteristic in normal condition and at elevated temperature equal to 700°C was studied. Strength of obtained materials are lower than nominal value of base material. Therefore for test performed in normal condition are equal to 690MPa and 610MPa at elevated temperature. Fracture of developed material are brittle with tendency to ductile in elevated temperature conditions. Heat treatment during tensile test affect on material properties, decreasing tensile strength with increase of elongation from value 4.5% up to 10%.

Microstructure of deposited material are dendritic and columnar dendritic. Solidification process are depended on deposition direction and affect in primal and secondary dendrites growth. Energy-dispersive X-ray spectroscopy analysis shown uniform mixture of deposited material, and no differences in chemical composition between substrate and deposited material. Quality analysis shown oxides inclusion in interlayer zone. No cracking, neither porosity was detected.

Manufactured material with laser engineered net shaping method have good strength characteristic. Sample measured at elevated temperature have greater plasticity than base material, however strength of deposited material in normal condition are greater. Plasticity of material testes in normal conditions are more than two times lower. Therefore material strength and elongation factor can be controlled via additional heat treatment, and machine components dedicated for work in high temperature conditions can be manufactured. However further research for developing machine components made from another type of nickel based super alloy as well as investigating properties of deposited material in higher elevated temperatures need to be performed. Moreover performing numerical simulation for better understanding process thermodynamics, where stress-strain analysis and temperature change over layers with analyzed thermal gradients in substrate and deposited materials are planned in further research [22].

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