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THE ANALISYS OF THE INFLUENCE OF COOLING RATE DURING LASER ALLOYING WITH SILICON NITRIDE ON SURFACE LAYER STATE OF CAST IRON MACHINE PARTS

Summary

The aim of this was to evaluate influence of different heat treatment conditions on microstructure and hardness of surface layer of cast iron elements. The molecular CO_2 laser with 2600W output power and TEM_{01} mode was used to perform surface modification. An optical and scanning microscopes, Auger electron spectroscope, X-ray diffractometer, EDS microanalyser and hardness Vickers tester were used to assess the result of the surface modification. The research showed, that it is possible to modify the surface layer of cast iron by laser alloying with silicon nitride. After laser alloying it is possible to achieve the alloyed zone (containing nitrogen and silicon) with uniform, fine, dendritic microstructure similar to the hardened white cast iron. Microstructure of alloyed zone as well as its size depended on laser heat treatment parameters. In case of alloyed zones formed with higher laser power density and its smaller interaction time (which generate higher cooling rates) it was noted higher amount of undiluted graphite and new-formed phases like $Fe_{1,94}C_{0,055}$, $FeN_{0,032}$, $FeN_{0,076}$, FeSi, Fe_2Si. In case of alloyed zone formed with higher cooling rate alloyed zone microstructure was finer and more homogenous. The average hardness of alloyed zone with silicon nitride was 5-times higher than matrix of the bulk material.
Improved hardness of surface layer of cast iron by laser alloying with silicon nitride should favor better wear resistance of machine part cast iron treated in this way.
***Key words**: laser alloying, silicon nitride, cast iron, surface layer*

ANALIZA WPŁYWU PRĘDKOŚCI CHŁODZENIA PODCZAS STOPOWANIA LASEROWEGO AZOTKIEM KRZEMU NA STAN WARSTWY WIERZCHNIEJ ŻELIWNYCH ELEMENTÓW MASZYN

Streszczenie

Celem badań była ocena wpływu różnych warunków laserowej obróbki cieplnej na mikrostrukturę i twardość warstwy wierzchniej elementów żeliwnych. Do modyfikacji powierzchniowej wykorzystano laser molekularny CO_2 o pracy ciągłej firmy Trumpf, o maksymalnej mocy 2600W i modzie TEM_{01}. Oceny przeprowadzonej modyfikacji dokonano za pomocą mikroskopu optycznego, skaningowego, spektroskopu elektronów Auger, mikroanalizy rentgenowskie oraz dyfrakcji rentgenowskiej, a także mikrotwardościomierza metodą Vickersa. Badania wykazały, że istnieje możliwość modyfikacji warstwy wierzchniej żeliwa za pomocą stopowania laserowego żeliw azotkiem krzemu. Po stopowaniu laserowym można uzyskać strefę stopowaną (zwierającą azot i krzem) o jednorodnej, drobnej, dendrytycznej mikrostrukturze, o charakterze zbliżonym do zahartowanego żeliwa białego. Mikrostruktura strefy stopowanej, jak i jej rozmiar zależały od zastosowanych parametrów laserowej obróbki cieplnej. W strefach powstałych z zastosowaniem większej gęstości mocy i krótszego czasu oddziaływania, generujących większą prędkość chłodzenia na materiał odnotowano większą zawartość nie rozpuszczonego grafitu, a także większą zawartość nowopowstałych faz jak: $Fe_{1,94}C_{0,055}$, $FeN_{0,032}$, $FeN_{0,076}$, FeSi, Fe_2Si. W przypadku stref uzyskanych z większą prędkością chłodzenia odnotowano większe rozdrobnienie i ujednorodnienie mikrostruktury. Średnia twardość stref stopowanych azotkiem krzemu była około 5-krotnie większa od twardości osnowy rdzenia. Zwiększenie twardości warstwy wierzchniej żeliw przez stopowanie laserowe azotkiem krzemu powinno sprzyjać zwiększeniu odporności na zużywanie obrobionych w ten sposób żeliwnych części maszyn.
***Słowa kluczowe**: stopowanie laserowe, azotek krzemu, żeliwo, warstwa wierzchnia*

1. Introduction

The automatization of agricultural production processes which containing area of agricultural production, as well as area of designing and construction of agricultural machines is one of the most important trend in agricultural technique development. Application of new materials and production techniques could cause crucial changes in machines construction. It is also connected to new methods of improving surface layer parts which are exposed i.e. to chemical and tribological loads [1].

This research concern surface treatment which could be apply in case of cast iron agricultural machine devices.

Cast irons are popular group of materials. They are used in many industry branches, also in agricultural industry [2]. Gray irons, particular nodular irons after appropriate treatment could replace cast steel or even steel elements. In some cases of parts working in soil like plough blades (usually made of 38GSA steel) for example ADI cast iron could be applied [3, 4].

Some parts of agricultural machines made of cast irons like: shafts of harvest machine, gears, teeth harrows, disc harrows, coulter presser feet are exposed to intensive wear. Consequently, appropriate surface layer properties are required. One of the surface treatment which allow to achieve changes in surface layer is laser heat treatment (LHT). Remelted surface layer achieve in nodular iron by this method could characterize by hardness increase comparing to the bulk material [5]. Cast iron after laser remelting or alloying (i.e. with boron) is characterized by better wear resistance, as well [6].

Except boron, other elements, like nitrogen could also be used in case of laser alloying. Nitrogen found spread application in diffusion treatment, mostly [7]. High hardness (i.e. 1200HV for low-alloy steel) of surface layer after nitriding could be achieve not only as a result of hard nitrides creation but also as a result of nitromartensite formation [8]. Results of research presented in [9-12] showed that high hardness, increase of wear resistance and corrosion resistance could be attain after laser alloying with nitrogen of steel parts. Laser alloying with nitrogen could be also reached using Si_3N_4 powder as a source of nitrogen [9]. During this research (concerning laser alloying of carbon steel) it was proved that decomposition of Si_3N_4 compound occurs. Additional advantage of this decomposition (except achieving nitrogen) is obtaining silicon which plays a great deoxygenate function.

Positive results after laser alloying with nitrogen of steel allow to expect good results after using this treatment in case of cast irons. In case of laser alloying it has to be take in to account the LHT parameters, particularly laser beam parameters, which have significant meaning in case of generating temperature of remelting zone and its heating and cooling rates.

The aim of this research was evaluation of influence of different laser alloying conditions with silicon nitride on microstructure and hardness of surface layer of cast iron.

2. Methodology

Laser alloying of nodular iron was performed with different laser beam parameters variants. Those selected variants cause different cooling rates of treated surface layer. Laser beam power was in range of 330÷2600W and its interaction time was in range of 0,2÷1,5s (laser beam diameter and laser beam fluence were constant). Those combinations allow to achieve different temperatures of remelted area and its different cooling rates which are responsible for microstructure and properties of modified surface layer. Due to applied laser beam parameters, theoretical cooling rate of treated surface was in range from 0,9 (for the lowest value of laser beam density and the longest interaction time) to 14,3 $\cdot 10^3$ $^oC/s$ (for the highest value of laser beam density and the shortest interaction time).

EN-GJS-600-3 nodular iron was selected as a test material. The chemical composition is presented in tab. 1.

Laser heat treatment consists of remelting of surface layer of cast iron. The surface was previously covered by appropriate coat coating alloying and bounding substance. Si_3N_4-α powder (with 325 mesh and purity $\geq$99,9% by Sigma-Aldrich) was used as the alloying substance (fig. 1).

Laser treatment was performed with the molecular CO_2 continuous Trumph laser type TLF 2600t at 2.6-kW output power and in the TEM_{01} mode.

Source: own work / Źródło: opracowanie własne

Fig. 1. Si_3N_4 powder applied during laser heat treatment
Rys. 1. Proszek Si_3N_4 stosowany podczas laserowej obróbki cieplnej

Results of the laser treatment were analyzed by means of a Zeiss Epiquant light microscope and Tescan Vega 5135 scanning electron microscope (zone geometry dimension evaluation and microstructure study), 3212 ZWICK Vickers microhardness tester with 100G load (microhardness distribution on the section of modified zones determination), D500 Kristalloflex Siemens X-ray diffractometer (phases identification), and PGT Avalon EDS microanalyzer and Auger LAS 620 RIBER (chemical compositions identification).

The research was performed at the Poznan University of Technology and at the University of Science and Technology in Krakow.

3. Results and discussion

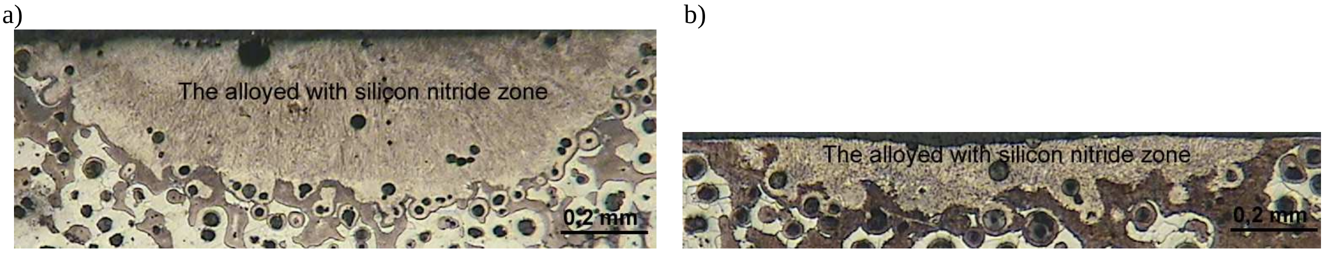
In each variant of laser treatment, the remelted (alloyed) zones, the hardened from the solid state and the transition zone between them were achieved (fig. 2). Existence of the hardened zone from the solid state is improving the durability of surface layer additionally. Furthermore, existence of transition zone is improving good bond between remelted zone and non-remelted bulk material.

The alloyed zone was characterized by fine and homogenous microstructure (especially in comparison to microstructure of the bulk material) in each laser treatment variant (fig. 2, 3).

Tab. 1. The chemical composition of EN-GJS-600-3 nodular iron
Tab. 1. Skład chemiczny żeliwa sferoidalnego EN-GJS-600-3

Cast iron	The element value % [wt.]								
	C	Si	Mn	P	S	Cr	Cu	Al	Mg
EN-GJS-600-3	3,50	2,87	0,32	0,03	0,013	0,03	0,559	0,009	0,038

Source: own work / Źródło: opracowanie własne



a) b)

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Fig. 2. The surface layer microstructure of cast iron after laser alloying with silicon nitride with laser beam power: P = 2600 W (a) and P =330 W (b). Optical microscope
Rys. 2. Mikrostruktura warstwy wierzchniej żeliwa po stopowaniu laserowym azotkiem krzemu z zastosowaniem mocy wiązki laserowej: P = 2600 W (a) i P =330 W (b). Mikroskop optyczny

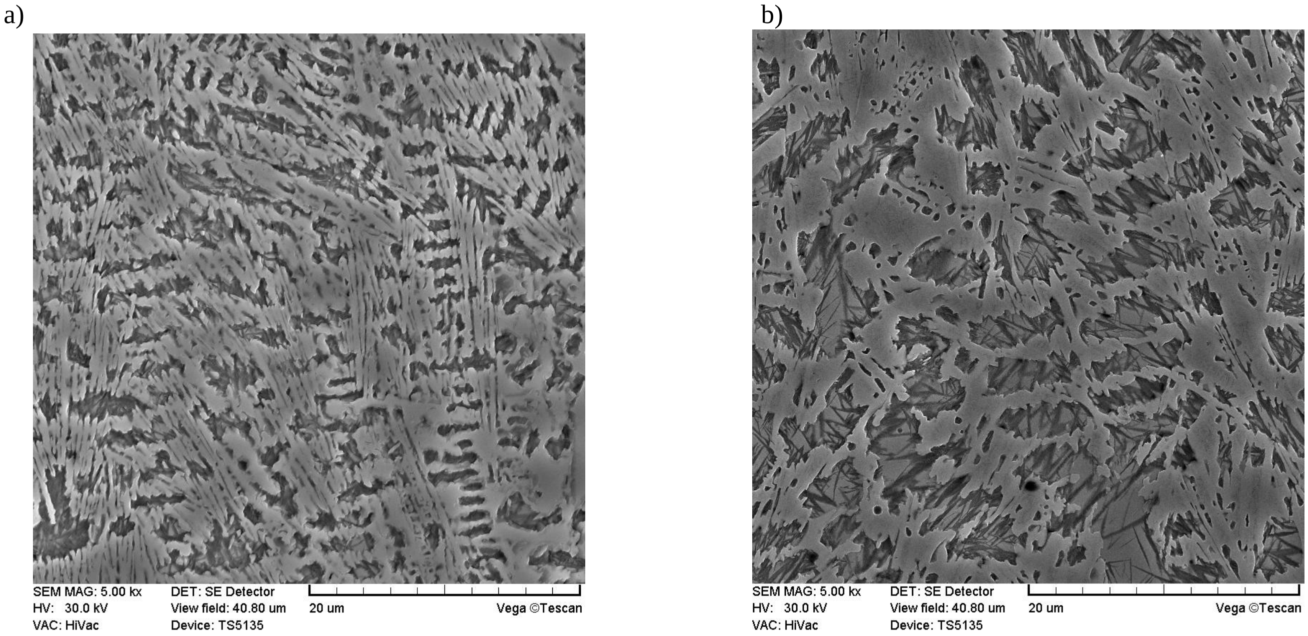

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Fig. 3. The part of microstructure of surface layer of cast iron after laser alloying with silicon nitride with laser beam power: P = 2600 W (a) and P =330 W (b). Scanning electron microscope

Rys. 3. Fragment mikrostruktury warstwy wierzchniej żeliwa po stopowaniu laserowym azotkiem krzemu z zastosowaniem mocy wiązki laserowej: P = 2600 W (a) and P =330 W (b). Elektronowy mikroskop skaningowy

It was noticed that, higher laser beam power density was applied, the bigger size of the alloyed zone was. This correlation was appeared in spite of shorter interaction time during application of higher laser beam power density. The size of cross section area S of alloyed zone in the surface layer of cast iron for different laser treatment variants is presented in figure 4.

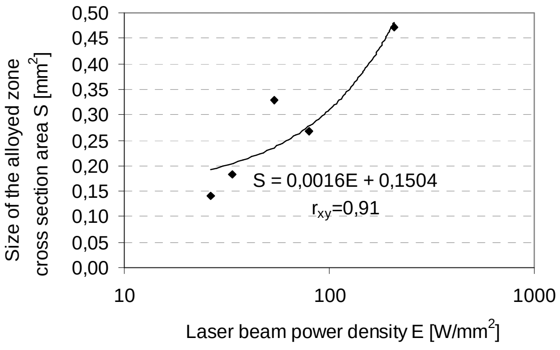

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Fig. 4. The influence of laser beam power density E on the size of cross section area S of alloyed zone in the surface layer of cast iron

Rys. 4. Wpływ gęstości mocy wiązki laserowej E na wielość pola przekroju strefy stopowanej S w warstwie wierzchniej żeliwa

The microstructure observation of alloyed zones except basic phases existing in Fe-C phase diagram did not revealed any additional inclusions (fig. 2, 3). All zones were dendritic. Their microstructure was similar to microstructure of the hardened white cast iron. Inside dendrites martensite needles were visible. These zones obtained with higher laser beam power densities (fig. 3) were characterized by finer microstructure (in comparison of zones obtained with lower laser beam power densities and longer interaction time). The average hardness of alloyed zones was 5-times higher than average hardness of matrix of the bulk material. No correlation between average hardness of alloyed zones and laser beam power densities was observed. Nevertheless, the zones obtained with higher laser beam power densities were characterized by smaller scatter of hardness. It confirms higher microstructure homogeneity in their cases.

Research by AES method confirmed the enrichment of the remelted zones with silicon nitride. AES method detected nitrogen and EDS method showed increased silicon amount in comparison to its amount in the bulk material. The temperature induced during LHT in the surface layer should makes possible decomposition of silicon nitride. This process was noticed during laser alloying with silicon nitride of steel [9, 11]. But, it is possible that nitrogen could also stay in compound with silicon because of higher affinity silicon to nitrogen than iron to nitrogen. X-ray diffraction research showed presence α and γ solid solutions, Fe_3C and $Fe_{1.94}C_{0.055}$ carbides. In the X-ray figures some peaks which are matching diffraction lines characteristic for $FeN_{0.032}$, $FeN_{0.076}$ and Fe_2Si, FeSi, $FeSi_2$ were also observed (nevertheless, it has to take in to account that some peaks make superposition). The example of the X-ray diffractograms for the alloyed zone and the bulk material were shown in the figure 5.

The diffraction research of phase composition in the alloyed zone allowed to assess relative values of amount of particular phases (fig. 6). But it should be taken into consideration that the value assess as ‘100’ indicate the alloyed zone which contains maximum amount of particular phase. Therefore, it needs to be emphasize that only values corresponding to the same phase could be compared.

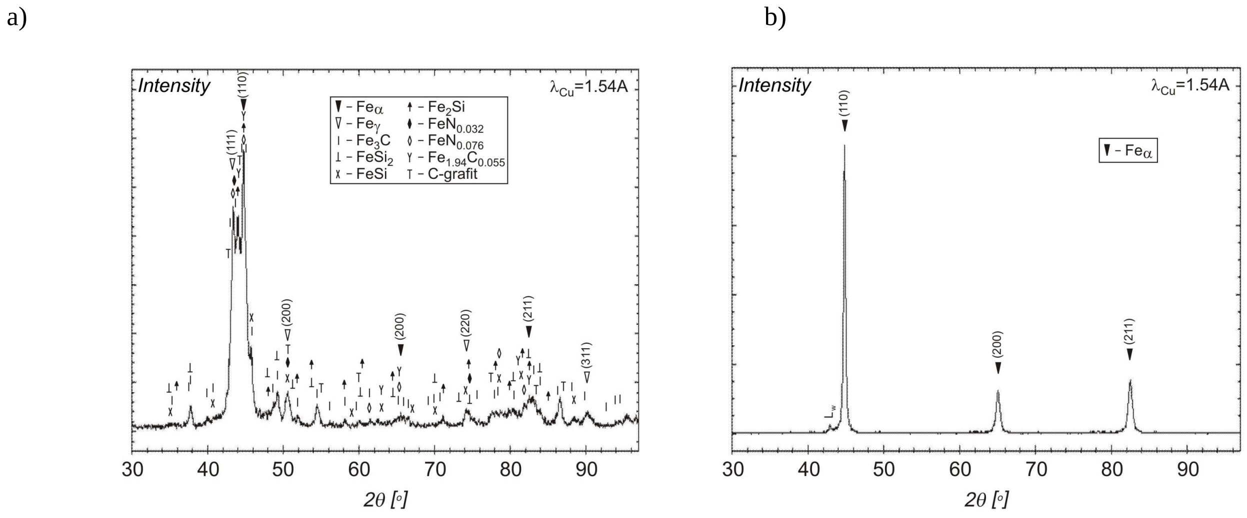

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Fig. 5. The example of the X-ray diffraction for the alloyed zone (a) and the bulk material (b)
Rys. 5. Przykład dyfraktogramów strefy stopowanej (a) i materiału rodzimego (b)

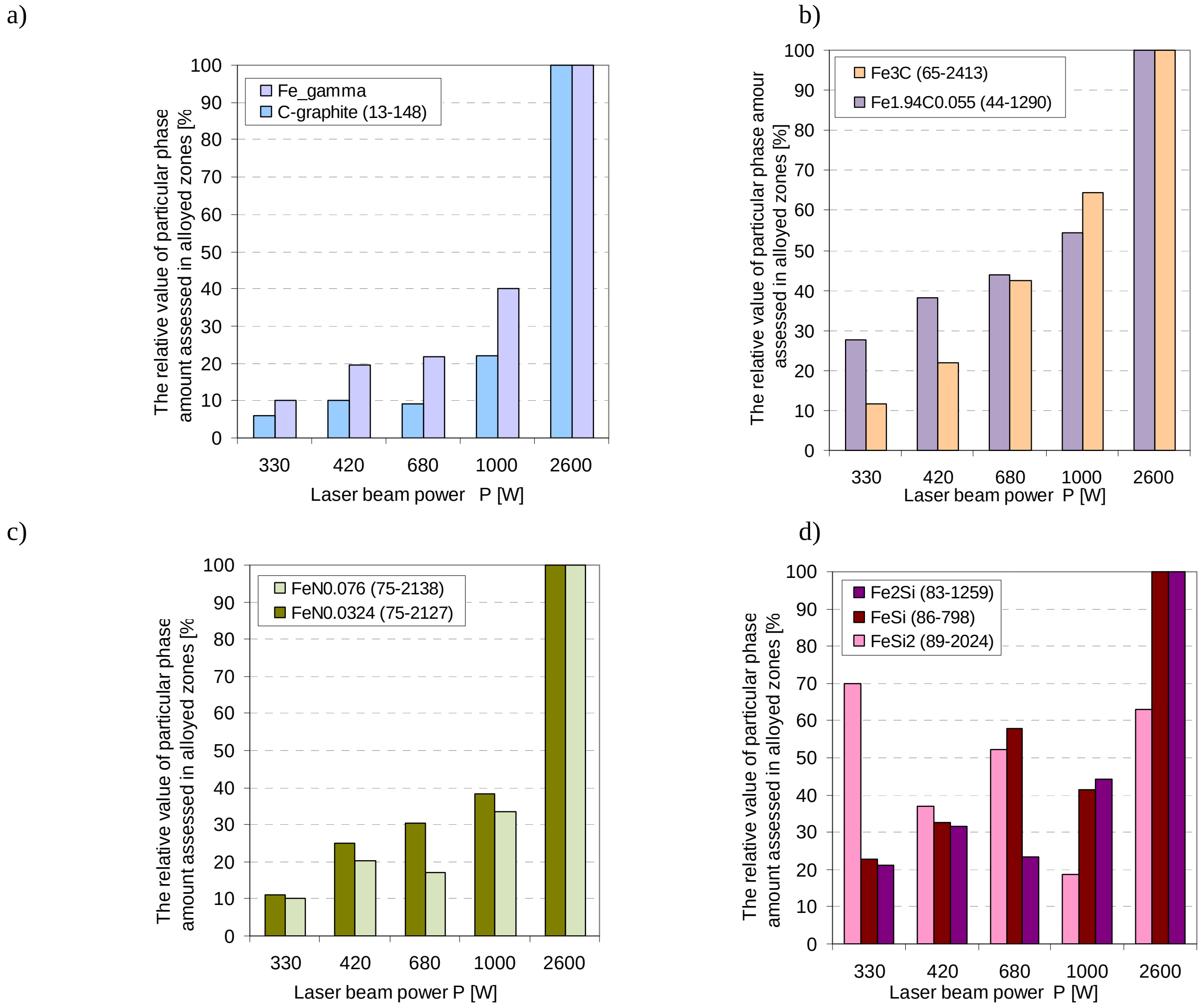

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Fig. 6. The relative value of particular phase amount assessed in alloyed zones after LHT with different laser beam parameters (a-d)
Rys. 6. Względna zawartość poszczególnej fazy oszacowana w strefie stopowanej po LOC z zastosowaniem różnych parametrów wiązki laserowej (a-d)

In case of higher laser beam density and shorter its interaction time to the surface application during LHT more graphite nodules could be observed in alloyed zone. It is due to limited time for diffusion. Carbon contained in graphite nodules does not have enough time to dissolve in liquid matrix during laser remelting (fig. 7).

Higher amount of Fe_3C, $Fe_{1,94}C_{0,055}$ and γ solid solution (fig. 8), as well as phases containing nitrogen: $FeN_{0,0324}$ and $FeN_{0,076}$ (fig. 9), and phases containing silicon: FeSi and Fe_2Si (fig. 10) could be expected in case of alloyed zones created in laser treatment conditions generating higher temperature and higher cooling rate of melting zone. Such microstructure conditions probably caused formation of more phases cells.

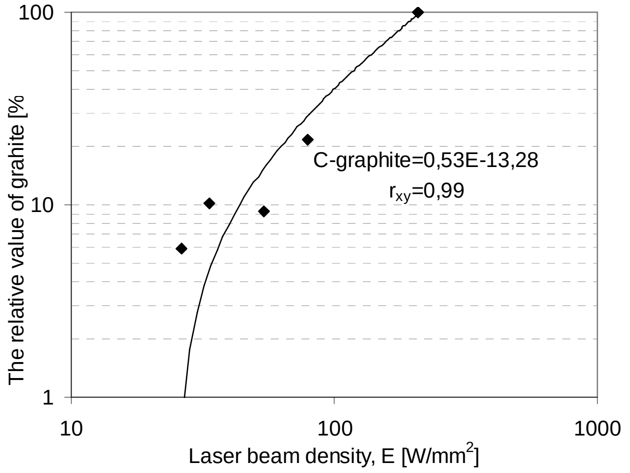

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Fig. 7. The relative value of graphite assessed in alloyed zone in case of different laser beam density application

Rys. 7. Względna zawartość grafitu oszacowana w strefie stopowanej w przypadku zastosowania różnych gęstości mocy wiązki laserowej

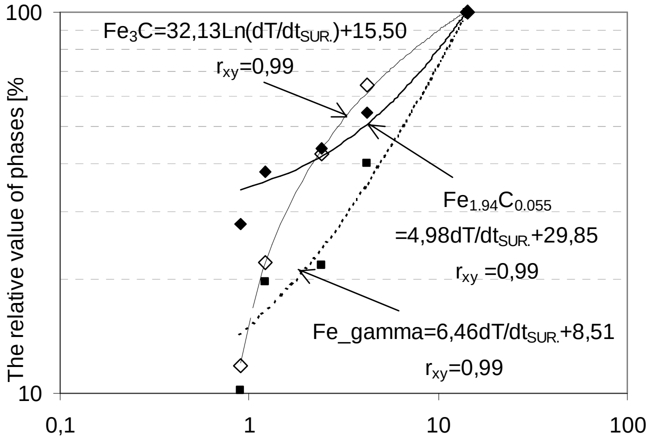

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Fig. 8. The relative values of Fe_3C, $Fe_{1,94}C_{0,055}$ and γ solid state assessed in alloyed zone with silicon nitride in the dependence of different cooling rates of remelted area in the surface layer of the treated cast iron

Rys. 8. Względna zawartość Fe_3C, $Fe_{1,94}C_{0,055}$ i roztworu γ oszacowana w strefie stopowanej w przypadku zastosowania różnych prędkości chłodzenia strefy przetopionej w warstwie wierzchniej obrabianego żeliwa

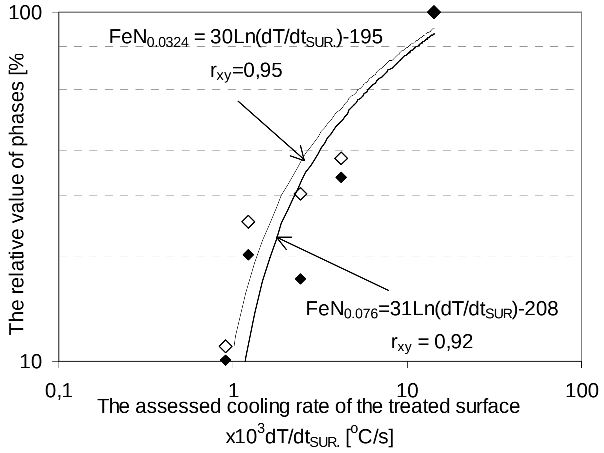

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Fig. 9. The relative values of $FeN_{0,0324}$ and $FeN_{0,076}$ assessed in alloyed zone with silicon nitride depending on different cooling rates of remelted area in the surface layer of the treated cast iron

Rys. 9. Względna zawartość $FeN_{0,0324}$ i $FeN_{0,076}$ oszacowana w strefie stopowanej w przypadku zastosowania różnych prędkości chłodzenia strefy przetopionej w warstwie wierzchniej obrabianego żeliwa

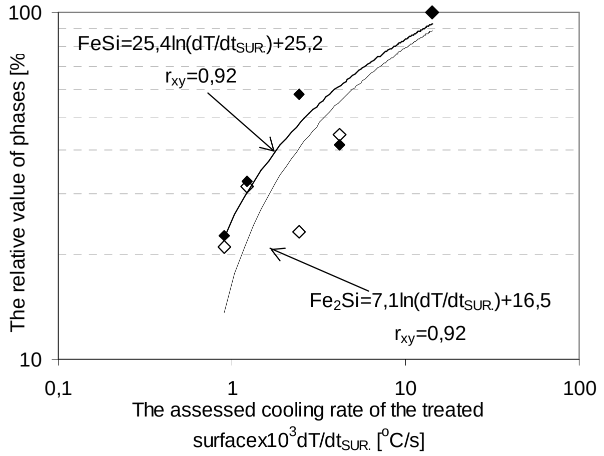

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Fig. 10. The relative values of FeSi and Fe_2Si assessed in alloyed zone with silicon nitride depending on different cooling rates of remelted area in the surface layer of the treated cast iron

Rys. 10. Względna zawartość FeSi i Fe_2Si oszacowana w strefie stopowanej w przypadku zastosowania różnych prędkości chłodzenia strefy przetopionej w warstwie wierzchniej obrabianego żeliwa

It means that LHT conditions determine microstructure of modified surface layer of cast iron significantly. Thus, by appropriate selection of LHT parameters it is possible to create proper microstructure of cast iron surface layer in case of alloying with silicon nitride.

4. Conclusions

On the basis on performed research following conclusions could be formed. It is possible to modify the surface layer of cast iron by laser alloying with silicon nitride. After laser alloying three zones in the surface layer could be selected:

– the remelted (alloyed) zone with uniform, fine, dendritic microstructure similar to the hardened white cast iron with 5-times higher hardness than matrix of the bulk material,

– the transition zone under remelted zone containing remelted and non-remelted phases during laser heat treatment,

– the hardened from the solid state zone under the transition zone.

Microstructure of alloyed zone as well as its size depended on laser heat treatment parameters. The higher laser beam density was applied, the size of alloyed zone was bigger, and its microstructure finer

In the alloyed zone nitrogen and higher amount of silicon then its amount in the bulk material was detected. The research showed, that except Fe-C phases like α, γ solid solutions, graphite, Fe_3C, and $Fe_{1,94}C_{0,055}$, phases containing nitrogen: $FeN_{0,0324}$ and $FeN_{0,076}$, and phases containing silicon: FeSi and Fe_2Si could appear.. In case of alloyed zones formed with higher laser power density and its smaller interaction time (which generate higher cooling rates) it was noted higher amount of undiluted graphite and new-formed phases. The higher amount of those phases could be a result of higher amount of phases cells created in alloyed zones cooling with higher rates.

In spite of the lack of correlation between average hardness of alloyed zones and laser beam power density, it was noticed that zones obtained with higher laser beam power densities were characterized by smaller scatter of hardness. It confirmed higher microstructure homogeneity in their cases.

The performed research showed that it is possible the microstructure modification and hardness increase of surface layer of cast irons by laser alloying with silicon nitride. Such treatment should improve wear resistance of the cast iron machine parts. It seems to be reasonable to perform wear tests of machine parts exposed to intensive abrasive wear like coulter presser feet laser alloying with silicon nitride.

It is worthy to emphasis, that by appropriate selection of laser heat treatment parameters it is possible to control creation of microstructure of alloyed surface layer, as well as, its size.

5. References

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