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Comparison of surface laser alloying of chosen tool steel using Al_2O_3 and ZrO_2 powder

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ABSTRACT

Purpose: In this work there are presented the investigation results of mechanical properties and microstructure of the hot work tool steel 32CrMoV12-28 alloyed with oxide powders like aluminium oxide and zirconium oxide. The purpose of this work was also to determine the laser treatment conditions for surface hardening of the investigation alloys with appliance of transmission electron microscopy.

Design/methodology/approach: The investigations were performed using optical microscopy for the microstructure determination. By mind of the transmission electron microscopy the high resolution and phase determination was possible to obtain. The morphology of the ceramic powder particles was studied as well the lattice parameters for the Fe matrix and phase identification using diffraction methods was applied.

Findings: After the laser alloying of the hot work tool steel with the selected oxide powders the structure of the samples changes in a way, that there are zones detected like the remelting zone the heat influence zone where the grains are larger and not so uniform as in the metal matrix. The used oxide powders are not present after the laser treatment in the steel matrix.

Research limitations/implications: The investigated steel samples were examined metallographically using optical microscope with different image techniques, SEM, TEM and analyzed using a Rockwell hardness tester, also EDS microanalysis and electron diffraction with Fourier transform was made.

Practical implications: As an implication for the practice a new technology can be possible to develop, based no diode laser usage. Some other investigation should be performed in the future, but the knowledge found in this research shows an interesting investigation direction.

Originality/value: The combination of TEM investigation for laser alloying of hot work tool steels makes the investigation very attractive for automotive and other heavy industries.

Keywords: Laser treatment; Surface treatment; Oxides; Tool steel

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1. Introduction

In this paper there is presented the laser treatment as a part technology applied for steel surface manufacturing. There is presented laser treatment with remelting of hot work tool steel 32CrMoV12-28 with ceramic powders especially oxides like aluminium oxide and zirconium oxide. The structure as well as improvement of mechanical properties is a practical aim of this work as well as improvement of hardness as a very important properties for practical use.

Nowadays, diode lasers do not merely deliver bits but also optical power. They are increasingly found in applications such as materials processing (welding, cutting, drilling, surface hardening, etc.) as well as in printing and graphical arts, in displays, and medical applications. In fact, since the advent of the high-power diode laser, laser technology is experiencing a fundamental structural change, as this semiconductor device has become the key element of a new breed of laser systems that are competing with gas lasers and lamp-pumped solid-state lasers. High-power diode lasers are continuously making inroads into industrial applications, as they are compact, easy to cool, yield a power efficiency beyond 50%, which is about five times higher than any other kind of laser has to offer (Fig. 1), and their costs are becoming increasingly attractive. To exploit the tremendous application potential (Fig. 2) of high-power diode lasers, research and development programs are performed in many industrial countries [1-8].

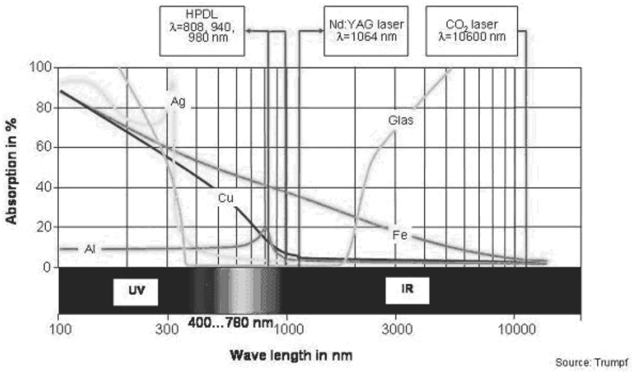

Fig. 1. Wave length range of the HPDL in comparison to Nd:Yag and CO_2 laser [Trumpf]

Most diode lasers are based on crystal wafers of group III-V compounds from the periodic chart of elements (Fig. 3). Those fabricated from gallium arsenide and its derivatives typically laser at wavelengths between 660 and 900 nanometer, and those utilizing indium phosphide-based compounds produce wavelengths between 1300 and 1550 nanometer. The dominant segment of the market is the shorter-wavelength lasers, with the most common application being in digital optical signal storage and retrieval, such as compact disc audio systems and read/write devices. The longer-wavelength diode lasers are primarily used by the telecommunications industries for fibre optic data transmission. Diode lasers can respond very rapidly, and in a linear fashion, to changes in driving current, which is a significant advantage in telecommunications transmission. The capability of a light source to be modulated at extremely high rates enables an enormous number of signals to be carried on a single optical fibre. As the technology surrounding the diode laser has evolved, dramatic improvements have been made in the efficiency, spectral characteristics, and functional lifetime of the devices. A primary objective in design of these lasers is preventing internal loss of radiation due to excessive beam spread from the small junction, where gain occurs. Through various techniques for confining the beam, not only is the efficiency and output power of the laser maximized, but also certain other characteristics of the beam are affected in a desirable manner [9-14].

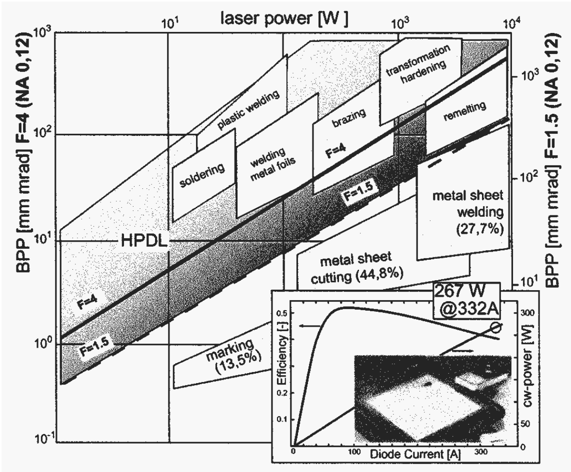

Fig. 2. Beam performance of HPDL in terms of laser power and beam parameter product are related to the required process intensity depending on application. The leading percentage (86%) of the present West European laser market for industrial applications in materials processing is indicated. The laser performance for two different F-numbers (F = 4 solid line, F = 1:5 dashed line) are given. A reasonable F-number (F = 1:5) allows for welding applications with commercial HPDLs. Commercial bars at 40 W, laboratory bars at 267 W

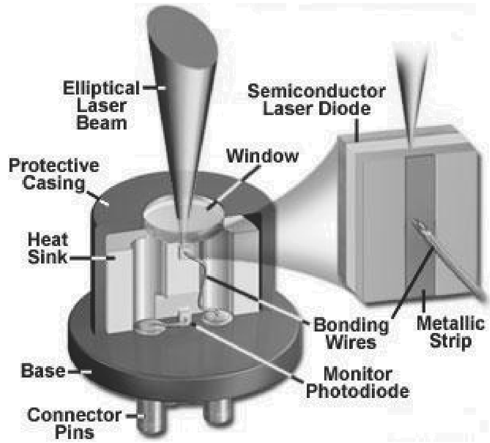

Fig. 3. Diode laser anatomy [Michael W. Davidson, The Florida State University]

One of the intrinsic HPDL properties is the rectangular beam shape, which can be profitably used in welding of large polymer parts. Polymer welding with a single moving HPDL beam was one of the earliest applications. Welding operations can be performed simultaneously when a single scanable beam is replaced by several larger rectangular beams. The rectangular beams are aligned along the whole weld track. For polymer-based windows, the length of the weld track is favorable for simultaneous weld processing as there are no moving parts. Beyond reduced handling costs, the residual mechanical properties of the weld governed by the thermal cycle can be controlled by the pulse shape and are no longer constrained by the welding speed. With increased pulse duration, also a larger lateral extent of the molten zone evolves and experimental experience showed improved adaptive gap bridging performance. In general, the additional degree of freedom for the processing parameters enables a more flexible process, and therefore investigation of new processes should be fruitful. An automotive component manufacturer successfully installed two 1.5-kW HPDLs for simultaneous hardening of the springs of door hinges. Flexible adaptation to a variety of geometrically different parts, minimized distortion by simultaneous hardening, and pyrometric monitoring of the quality are additional features to these low-cost and high-productivity laser system. Cladding of engine valves out of nimonic with stellite clad material is demonstrated using a 2.5-kW HPDL. The required width of the clad track, 3 mm, and a depth of about 1 mm are achieved [1]. Relatively low intensities in the range of Wcm are sufficient for these applications. Experimental results on metal working with higher intensities up to Wcm were obtained using industrial HPDLs at proper focusing conditions with an output power of 1.3 and 2.5 kW, respectively. While these HPDLs are built on the basis of commercial diode laser bars with 40 W output power each, in the meantime a record continuous-wave output power of 267 W per bar has been demonstrated in the laboratory [4, 15-18].

The purpose of this work is to study the effect of a HPDL laser melting on the hot work tool steel, especially on their structure and hardness. Special attention was devoted to monitoring of the layer morphology of the investigated material and on the particle occurred.

2. Experimental procedure

The material used for investigation was the hot work tool steel 32CrMoV12-28, it has been supplied annealed in a form of rods 76 mm in diameter and in the length of 3 m. Of this material samples 70 x 25 x 5 mm were cut off. The chemical composition of the investigated steel is presented in Table 1.

The samples were heat treated according to the normalized steps for this type of steel, at first tempering was performed and then annealing. Austenisation was performed in a vacuum furnace at a temperature of 1040 ºC, the tempering time was 0.5h. During the heating to the austenitic temperature two izothermic holds were applied, the first in the temperature of 585 ºC and the second at 850 ºC. After tempering two annealing operations were performed during 2h each, the first in 550 ºC and the second in 510 ºC. After heat treatment the surface of the samples were grinded on a magnetic grinding machine. Special care was set to avoid of the micro cracks, which can disqualify a sample for future investigation. On the so prepared and fatless samples the used powders was carried on. The powder was before mixed with inorganic sodium glass in ratio 30% glass and 70 % powder. Every time a paste layer of 0.5 mm in thickness was carried on. Based on the investigations performed during remelting using the high power laser diode HPDL Rofin DL 020 with work parameters presented in Table 2. The maximum speed for a good work process is v = 0.5 m/min. For this reason all the investigations were carried out in a constant remelting process speed, changing the laser power in a range of 1.2 - 2.3 kW. By laser power of 0.4 - 0.8 kW there are not remelted areas present at all.

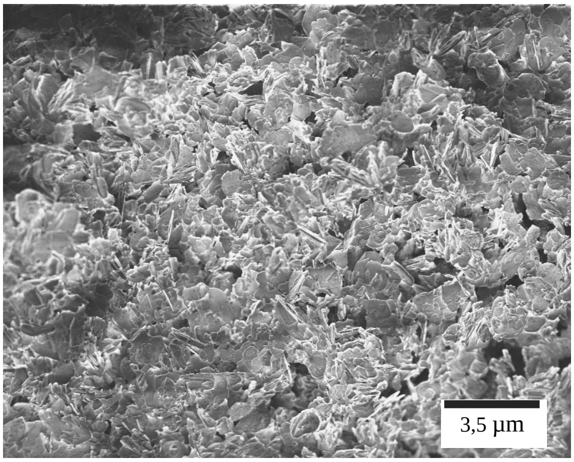

Fig. 4. ZrO_2 powder used for alloying

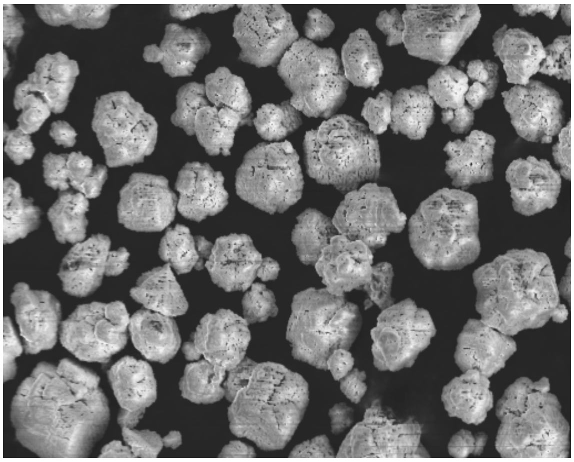

Fig. 5. Al_2O_3 powder used for alloying

Table 1.
Chemical composition of the investigated hot work tool steel 32CrMoV12-28

	Mass concentration of the elements, %			
steel 32CrMoV 12-28	C	Si	Mn	P
	0.308	0.25	0.37	0.02
	S	Cr	Mo	V
	0.002	2.95	2.70	0.535

The samples were mounted in the laser holder for remelting. On each sample surface four laser process trays were performed of a length of 25 mm, according to the power 1.2; 1.6; 2.0; 2.3 kW. It could be set experimentally, that the fully protection of the remelted place can be achieved by mind of an argon protective atmosphere with a gas flow rate of 20 l/min through a round nozzle with a diameter of 12 mm, which was directed inversely to the remelting process direction.

Table 2.
HPDL laser parameters

Parameter	Value
Laser Wave lenght, nm	940 ± 5
Peak power, W	100 ± 2300
Focus length of the laser beem, mm	82 /32
Range of power density of the laser beam In the focus plane, kW/cm²	0.8 - 36.5
Dimensions of the laser beam focus, mm	1.8 x 6.8

Aluminium oxide Al_2O_3 and zirconium oxide ZrO_2 powders were used for alloying (Figs. 4 and 5). The powder was put to the so prepared and degreased samples. The powder was initially mixed before with the inorganic sodium glass in proportion 30% glass and 70 % powder. A paste layer of 0.5 mm in thickness was put on. The average granulation of the applied powders was in the range of ca 10 µm. Some other physical parameters of the applied powders are presented in Table 3.

Table 3.
Chosen physical parameters of the used oxide powders

Properties	Al_2O_3	ZrO_2
Hardness, HV	2300	1300
Density, kg/m3	3.90-3.99	5.56
Thermal expansion coefficient, 10-6.K-1	7.4-8.5	5-10
Melting temperature, °C	2047	2715
Granulation, µm	10	6

Structure investigation and phase identification was performed using transmission electron microscopy (TEM) JEOL JEM 3010 as well scanning electron microscope (SEM) together with energy dispersive spectroscopy (EDS). The hardness was measured with Rockwell hardness tester with a load chosen for the HRC scale. A minimum of 10 indentations was made on each of the tested samples. Phase composition and crystallographic structure were determined by the X-ray diffraction method.

3. Results and discussion

The micrographs of the microstructures and structure investigation was performed using the light microscope Leica MEF4A supplied by Zeiss in a magnification range of 50 - 500x. The micrographs of the microstructures were made by means of the KS 300 program using the digital camera equipped with a special image software. Metallographic investigations and the observations were performed on the cross section (Figs. 8, 9 and 10), where the laser treatment thickness can be measured and the structure can be revealed in this case for the Al_2O_3 powder alloyed samples using 1.2 kW laser power. The structure of the remelted zone is a dendritic one. Roughness investigation of the sample on each of the remelting trays were carried out, the surface structure is presented on Figs. 6 and 7.

For each remelting area Hardness measurements results were registered, for this reason the Rockwell hardness tester supplied by Zwick was used according to the PN-EN ISO 6507-1 standard, by a load of 147.2 N for 15 s. TEM investigation results are showed on Fig. 15. TEM investigations were performed on the JEOL JEM 3010 microscope using bright and dark field image technique and SAD method. The diffraction pattern calculation was performed using the “Eldyf” software supplied by the Institute of Material Science o the University of Silesia. Diffraction pattern analysis of the investigated areas has allows it to identify the Feα matix (Fig. 11) as well the cubic NbC phase of the 225 - Fm-3M space group with the [112] zone axis (Fig. 12), the high resolution image with Moiré fringes (Figs. 13 and 14) together with the FFT diffraction image can be used to calculate the space parameter d of the Fe matrix. The fringes itself give a space distance of 4.4 Angstrom so it is not the real lattice level, but it can be calculated using this data.

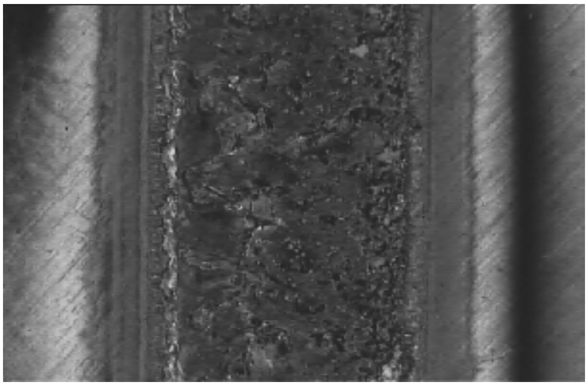

Fig. 6. Shape of the laser tray of the 32CrMoV12-28 steel remelted with Al_2O_3 powder, laser power 2.3kW

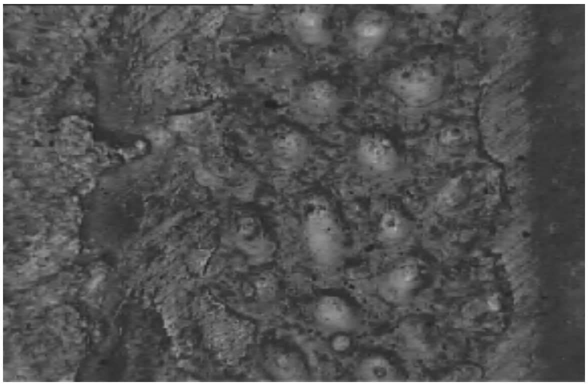

Fig. 7. Shape of the laser tray of the 32CrMoV12-28 steel remelted with ZrO_2 powder, laser power 1.2 kW

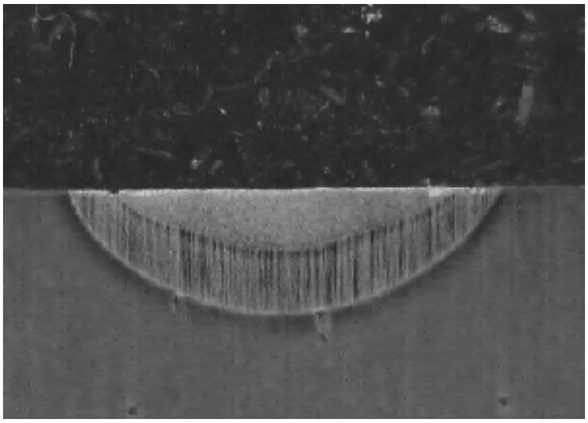

Fig. 8. Shape and thickness of cross-section of the laser remelted samples with Al_2O_3 laser by power of 2.3 kW

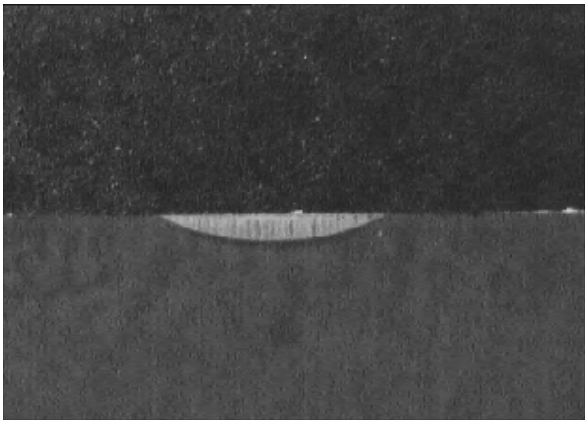

Fig. 9. Shape and thickness of cross-section of the laser remelted samples with ZrO_2 laser by power of 1.2 kW

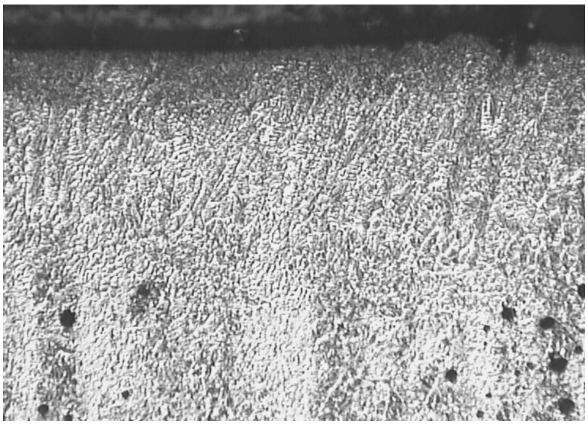

Fig. 10. Structure of the surface layer after remelting with Al_2O_3 powder using 1.2 kW laser power

a)

b)

c)

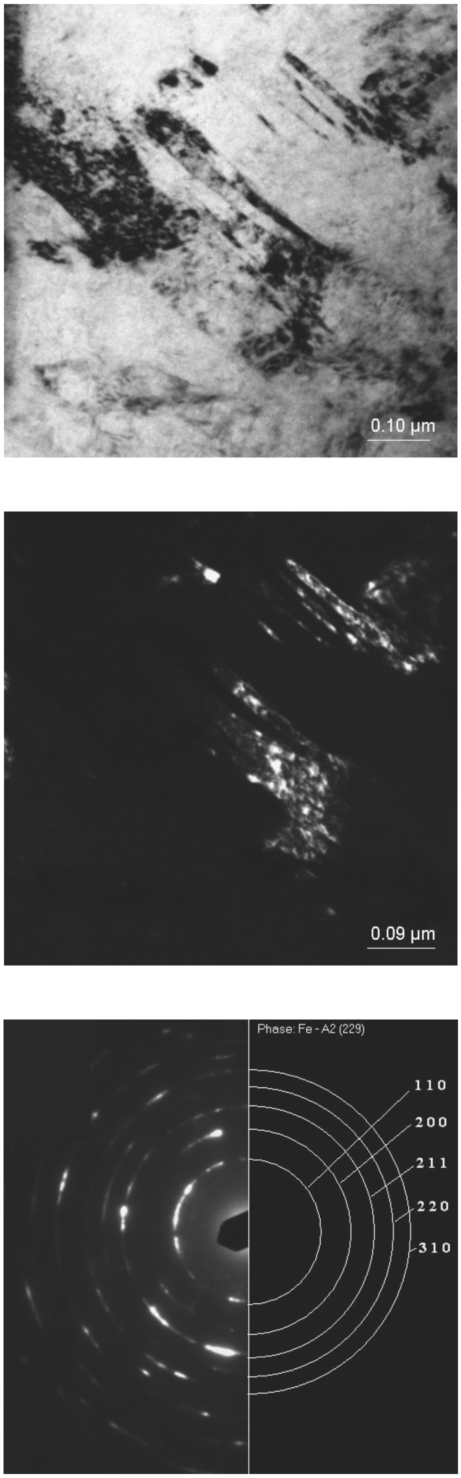

Fig. 11. TEM microstructure, a) bright field image, b) dark field image, c) diffraction pattern and its solution of the area in Fig. 13a

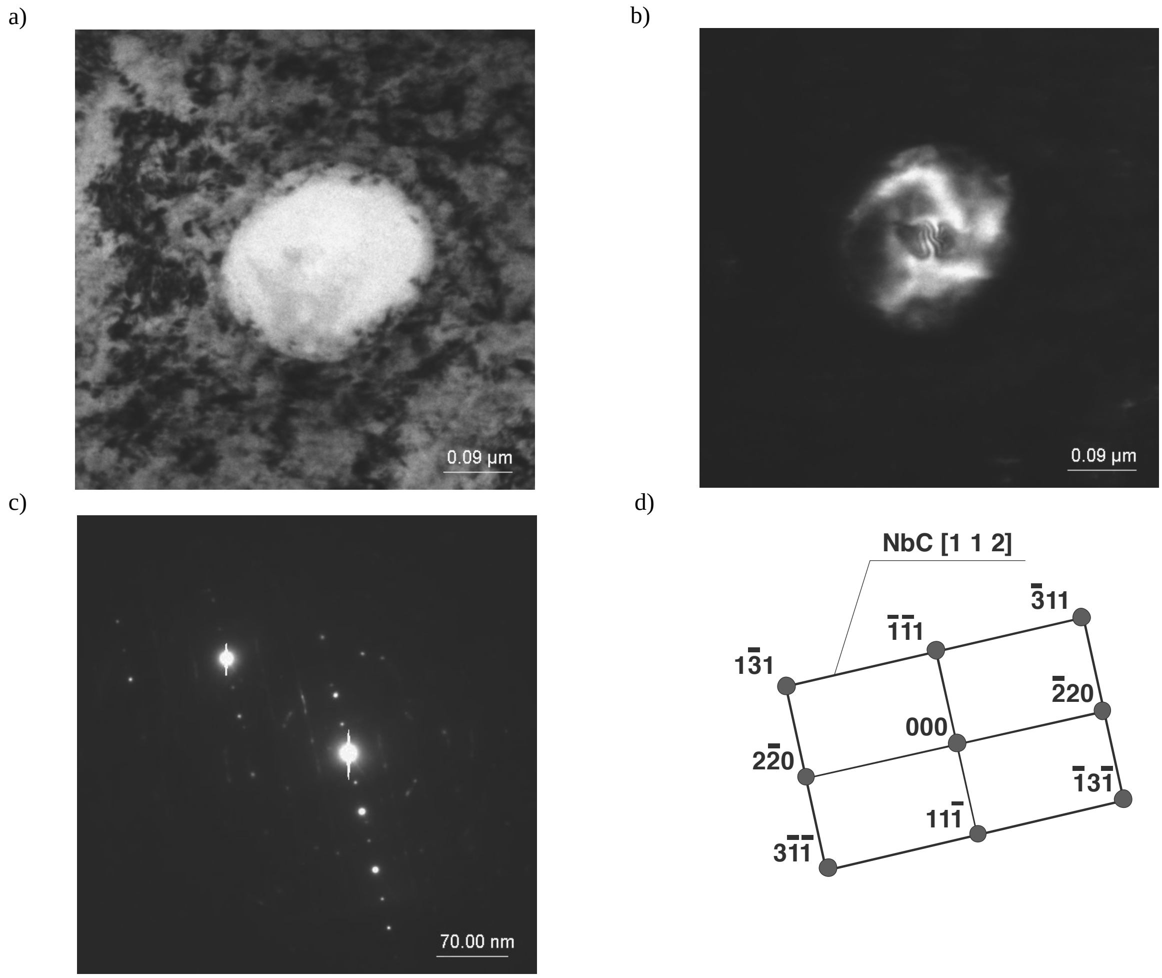

Fig. 12. TEM microstructure, a) bright field image, b) dark field image, c) diffraction pattern of the particle in Fig. 2a, solution of the diffraction pattern in Fig. 2c, high resolution image

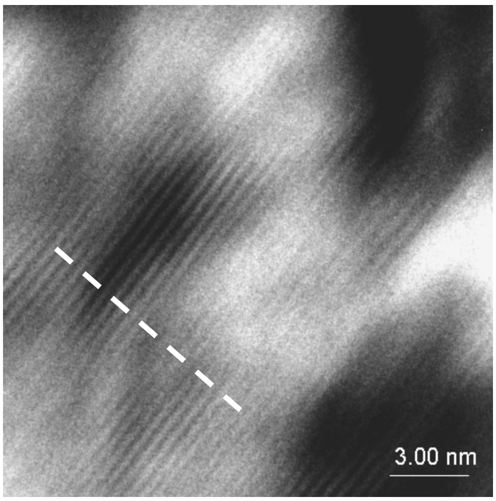

Fig. 13. High resolution TEM bright field image

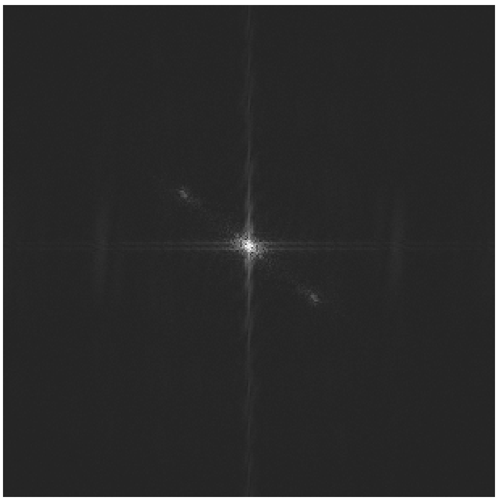

Fig. 14. FFT Diffraction pattern image

The required hardenability for this tool steel was achieving after a suitable tempering time, which assures melting of the alloying carbides (Fig. 12) in the austenite. The structural investigations carried out using the high power diode laser allows to compare the surface layer as well as the shape and depth of the remelting area.

The layers achieving by the alloying process are showed on Figs. 6-9. The results allows to state that with the increasing laser power the roughness (Figs. 6 and 7) of the remelted metal surface increases. Preliminary investigations of the remelted hot work tool steel 32CrMoV12-28 show a clear effect of the laser power respectively 1.2; 1.6; 2.0 and 2.3 kW on the shape and thickness of the remelted material (Figs. 8 and 9). Microstructure presented on Fig. 10 shows a dendritic structure in the remelted area, but there are no evidence of the powder particles used for investigation distributed in the matrix. There is also a clear relationship between the employed laser power and the dendrite size, namely with increasing laser power the dendrites are larger. The hot work tool steel has a ferritic structure with homogeny distributed carbides in the metal matrix in the annealed state. In areas, which are between the solid and molten state dendritic structure with large dendrites can be found.

The results of the TEM diffraction analysis shown in Fig. 11 confirm the lathe martensite as the Feα matrix, as well the occurrence of NbC particles in the steel matrix. But there is no evidence for the used oxide powder particles distributed in the steel after laser. Future scanning microscope structural investigations should reveal the occurrence of the used oxide particle or denial it.

As a result of laser alloying the difference of the remelted area grain size (Fig. 16) among the power of 1.2 kW and 2.3 kW is about 4-5 times larger for the 2.3 kW power for both powders used. Fig. 15 shows the hardness measurements results of the remelted surface for 1.2, 1.6, 2.0 and 2.3 kW laser power. The highest hardness value is achieved for the 2.3 kW laser power for the zirconium oxide powder, and decreases with laser power for the aluminium oxide powder.

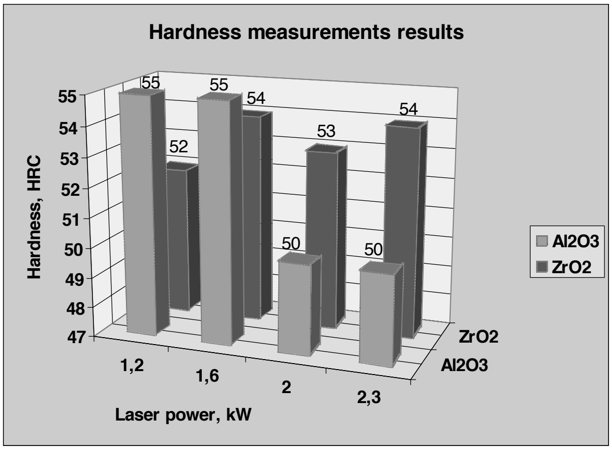

Fig. 15. Hardness measurements results of the Al_2O_3 and ZrO_2 alloyed steel

The highest value is achieved for alloyed top surface - the remelted zone and decreases with the remelting depth through the heat affected zone to the hardness value of the steel matrix is achieved.

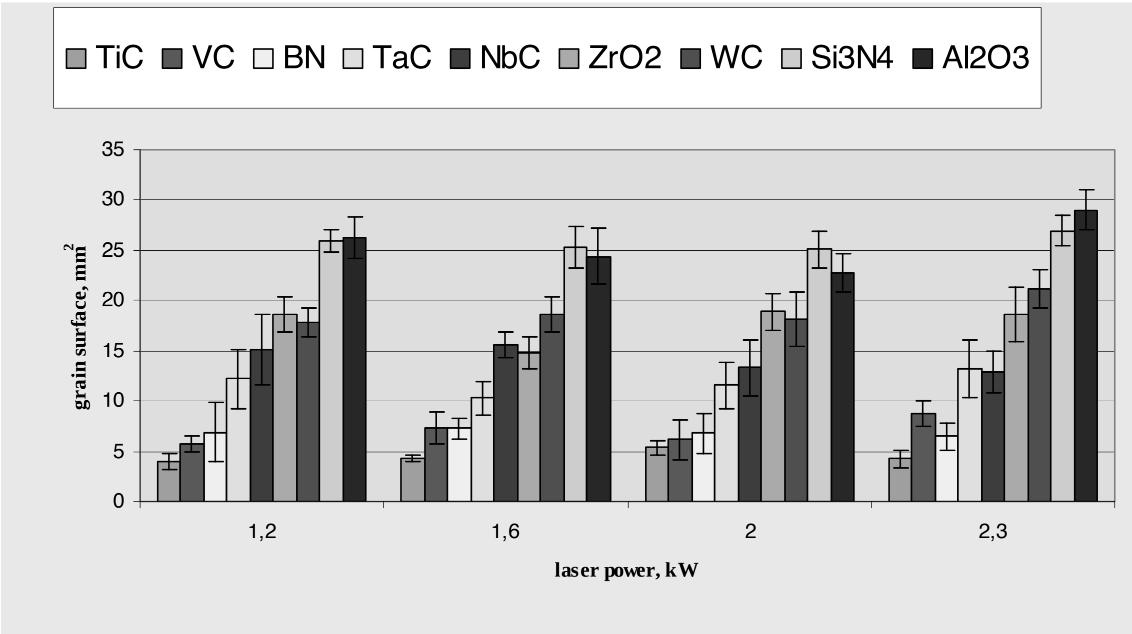

Fig. 16. Average grain surface area of the remelted zone SP of the 32CrMoV12-28 steel alloyed with ceramic powders wit laser power in the range of 1.2 to 2.3 kW

4. Conclusions

The investigations in this work makes it possible to conclude, that as a result of laser alloying as well as remelting of the hot work steel 32CrMoV12-28 with the ceramic powder, especially oxides, but also carbides and nitrides a suitable surface layer of high-quality is possible to achieve. The surface layer in case of alloying with oxides contain no cracks and defects as well is of much more higher hardness value compared to the material which was not alloyed and only laser-remelted. In case of Al_2O_3 ceramic oxide powder the increasing laser power depth of remelting material is higher compared to ZrO_2 and the surface is more regular. What is the most important thing, the hardness of the surface layer is the highest among all the others ceramic oxide powder used, but only for 1.2 and 1.6 kW laser power. Here the hardness value does not increase according to the laser power used, so that the lowest power applied gives to highest hardness value in the remelted layer. Only in case of zirconium oxide powder the hardness of the surface layer increases with increasing laser power used. For the Al_2O_3 the surface of the remelted area is more regular, less rough and more flat with increasing laser power. The investigations on scanning and light microscope reveal a dendritic structure in the remelting zone in samples alloyed with every applied laser power. The dendrite size increases with the increased laser power. Also the wear resistance of the surface layer of the worked steel increases with increased laser power. The resistance increases also compared to the steel remelted only, that confirms the use of ceramic powder as a material for improvement of mechanical properties. Transmission and scanning electron microscope investigation do not allow to reveal the oxide powders used for alloying, so there is a possibility that the powders are at least partially solute in the steel matrix, but this should be a matter of next investigations.

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DEVELOPMENT FUND

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