Effect of Diode Laser Surface Alloying of Hot-Work Tool Steel

L. A. Dobrzanski, M. Bonek, E. Hajduczek, A. Klimpel

The objective of the present work was to study the modification of the microstructure of hot-work tool steel X40CrMoV5-1 during the surface modifying by means of laser technology.
The aim of such treatment was to harden and alloy the steel surface which had been previously coated with tungsten carbide (WC). Development of the surface layer was observed in which one can distinguish the remelted zone, heat-affected zone and the transient zone. The fine grained, dendritic structure occurs in the remelted and alloyed zone with the crystallization direction connected with the dynamical heat abstraction from the laser beam influence zone. The fine grained martensite structure is responsible for hardness increase of the alloyed layer.

Key words: hot-work tool steel, surface layer, gradient coating, remelting, alloying, high power diode laser

INTRODUCTION

The moulding cast allows the performance of a lot of quantity of components with high-level complexity, fulfilling the mechanical specifications required, with a medium-low relative prices.
The material of our study is steel used to make a lot of components of mechanized, as dies for moulding cast for example. The design and the service-behaviour of the steel die are the most important factors in order to achieve the required specifications in moulding cast. These factors not only control the final dimensions and tolerances but even the surface finish. The thermal stress producing by hot-cooling cycles promotes little cracks which make the life of the die shorter. One die costs almost one million dollar and needs from months to years to be made. Improvements in the steel make better final components, but also a longer life of the die. If the die has a longer life, the costs will be lower and also the energy consumption. The hot-work steel X40CrMoV5-1 is one of the most used steels to make dies because of its good properties in thermal fatigue, corrosion and wear away resistance. That is why the hot-work tool steel X40CrMoV5-1 is very used to cast aluminium, magnesium and their alloys [1-7], [12].
Figure 1 shows how is applied the laser technology in the automotive industry for different applications. The main advantages of using laser technology in this industry are: More versatility to applications with more diversity if we compare the dimensions and the kind of material, the thermal effect over the simple is lower, the process quality is very high, very high velocity of production, easy to integrate into robotic systems. In the Others (4%) sector are included the laser surface treatments [11].
The investigations about surfacing-alloys reveal that the surface treatments and the surface coatings are very good in order to protect the dies against the thermal fatigue, and increase the life of the die too. In fact, the surface coatings in-
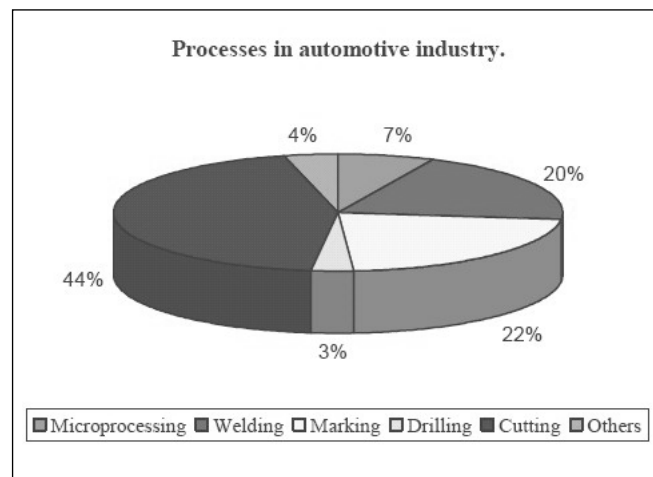
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Fig. 1 – Sector distribution in the processing of materials by laser meanings [11, 12].

Fig. 1 – Incidenza delle diverse tecniche di trattamento dei materiali con mezzi laser [11, 12].

crease the resistance to abrasion because it provides higher hardness, toughness and corrosion resistance [15].
One of this treatments could be laser surface treatment, which allows modify the properties of the surface of the material without modifying the core of it. These kinds of treatments make a very low grain and homogenous microstructure at the surface with a very short HAZ (Heat Affected Zone), providing a higher solid solubility by the alloying elements. The resulting surface has very good mechanical properties because the steel is melting and cooling in a very short period of time ($10^4 – 10^5$ times faster than conventional moulding), giving rise to a composition, distribution of the alloying elements and microstructural changes [8-10]
Employment of the laser surface treatment is justified both from the economical point of view and because the laser treatment, in many cases, ensures obtaining better mechanical properties of the processed surfaces, e.g., teeth of gear wheels or cutting tools edges, which could not be attained using the conventional surface treatment methods [13, 16-22]. The laser heat treatment includes operations which are conducted using the laser beam as the source of energy needed for heating the surface layer of the processed material, to change its structure for obtaining the relevant mechanical, physical, or chemical properties, improving service life of the processed element. [14]

EXPERIMENTAL PROCEDURE

The experiments were made on specimens made from the X40CrMoV5-1 hot work tool steel. The chemical composition of the steel is presented in Table 1. The investigated steel was molten in the electric vacuum furnace at the pressure of about 1 Pa, cast into ingots weighing about 250 kg, and were roughed at the temperature range 1100-900ºC into the O.D. 75 mm bars, which were soft annealed. After making by machining the O.D. 70 mm and 6 mm thick specimens they were heat treated. The specimens were austenitized on the salt bath furnace and tempered in the chamber furnace in the protective atmosphere – argon. The specimens were gradually heated to the austenitizing temperature with the isothermal stops at 650 and 850°C for 15 min. Further they were austenitized for 30 min at the temperature of 1060°C and cooled in hot oil. The specimens were tempered twice for 2 hours at the temperature of 510°C after quenching. Surfaces of specimens were sand blasted and machined on magnetic grinder. Next, powder layers of the WC tungsten carbide of two different thicknesses of 0.06 mm and 0.11 mm bounded with the inorganic binding agent were put down onto the degreased specimens. The siliceous liquid glass consisting of the Na_4SiO_4 orthosilicate and $Na_2Si_2O_5$ sodium disilicate was used as a binding agent in the form of the syrup like solution obtained under the increased pressure. Selected properties of the commercial tungsten carbide, being a mixture of the WC tungsten carbide and pure tungsten are presented in Table 2. Specimens of the X40CrMoV5-1 steel fixed in a turntable were remelted with the Rofin DL 020 high power laser beam (HPDL) with parameters specified in Table 3. The dimensions of the laser beam focused on the material surface are 1.8 x 6.8 mm. Remelting was carried out perpendicularly to the longer side of the focused beam with the multimode energy distribution, which makes it possible to obtain the wide run face. The working focal length (measured from the protective glass in the head) is 92 mm. The multimode energy distribution was

Average mass concentration of elements, %								
C	Mn	Si	Cr	W	Mo	V	P	S
0.41	0.44	1.09	5.40	0.01	1.41	0.95	0.015	0.010

Table 1 – Chemical composition of X40CrMoV5-1 steel.

Tab. 1 – Composizione chimica dell'acciaio X40CrMoV5-1.

Average grain diameter, µm	Melting temp., °C	Density, g/cm³	Hardness, HV30
20 - 30	2730 - 2870	15.6	1550

Table 2 – Selected properties of the WC powder.

Tab. 2 – Alcune caratteristiche della polvere di WC.

Wavelength of the laser radiation, nm	808 ± 5
Maximum output power of the laser beam (continuous wave), W	2500
Power range, W	100-2500
Focal length of the laser beam, mm	92
Laser spot size, mm	1.8 × 6.8
Power density range in the laser beam focal plane, kW/cm2	0.8-36.5

Table 3 – Technical date for the HPDL ROFIN DL 020.

Table 3 – Dati tecnici del materiale HPDL ROFIN DL 020.

used. It was found out in the preliminary investigations that the maximum feed rate at which the process is stable is 0.5 m.min. Further experiments were carried out at the constant remelting rate, changing the laser beam power in the 0.5-1.9 kW range during alloying the test pieces. It was established experimentally that the argon blow-in with the flow rate of 20 l/min through the φ 12mm circular nozzle oppositely directed in respect to the remelting direction provides full remelting zone protection. The test pieces were machined after remelting and alloying, to remove the non-remelted layer of the tungsten carbide.

The microsections' surfaces were ground on diamond wheels and next polished using the diamond buffing compounds on Struers equipment. Etching of the specimens was carried out in nital with the following composition: 2-5 ml HNO_3 nitric acid, 100 ml C_2H_5OH ethyl alcohol, at room temperature. Etching time was selected experimentally for each investigated material's surface layer form. Metallographic examinations of material structure after laser alloying of its surface layer were made on Leica MEF4A light microscope at magnifications from 100 to 1000x. The Leica – Qwin computer image analysis system was used for thickness examination of the particular zones of the surface layer and for measurement of grain areas and dendrite lengths. Grain size analysis was carried out on the steel microstructure images saved in the electronic form. The stored examination results of the average grain size and dendrite lengths in the particular zones were analysed statistically. Structure examinations and thickness measurements of the relevant zones in the surface layer were made also for comparison on the transverse microsections on the Opton DSM 940 scanning electron microscope at magnifications 1000 and 5000x. The X-ray qualitative and quantitative micro-analysis and analysis of the surface distribution of the alloying elements in the test pieces of the investigated steel, subjected to the standard heat treatment and remelted and alloyed, were made on the Opton DSM-940 scanning electron microscope with the Oxford EDS LINK ISIS X-ray energy dispersive spectrometer at the accelerating voltage of 20 kV and on the JEOL JCXA 733 X-ray micro-analyser. The crystalline X-ray wavelength dispersive spectroscopes (WDS) were used for quantitative analysis, using the accelerating voltage of 20 kV, and 10 kV during carbon analysis. Thin foils were made from lamellae diced on the electrodischarge machine from the test pieces for the structural examinations, from which disks with 3.2 mm O.D. were cut out, next mechanically thinned and ion polished in the electrolyte on the Struers TENUPOL-2 electrolytical polishing device and on the Gatan 691 ion polishing device. Diffraction and chemical composition examinations in micro-areas and of the thin foil structures were made on the JEOL 200CX transmission electron microscope at the accelerating voltage of 200 kV, equipped with the Oxford EDS LINK ISIS X-ray energy dispersive spectrometer and on the TESLA BS 540 electron microscope at the accelerating voltage of 120 kV. The diffraction patterns from the transmission electron microscope were solved using the computer program. Analysis of the crystallographic relationships occurring between the phases identified in the diffraction patterns from thin foils was made using the stereographic projections.

ANALYSIS OF EXPERIMENTAL RESULTS

When laser is applied to one metal surface, this surface absorbs the energy of the laser radiation.
The absorbed energy becomes in heat, rising the temperature of the metal (but only in the surface).
The depth of the zone affected for this radiation depends on the laser power and on the absorbance of the surface [14]. La-
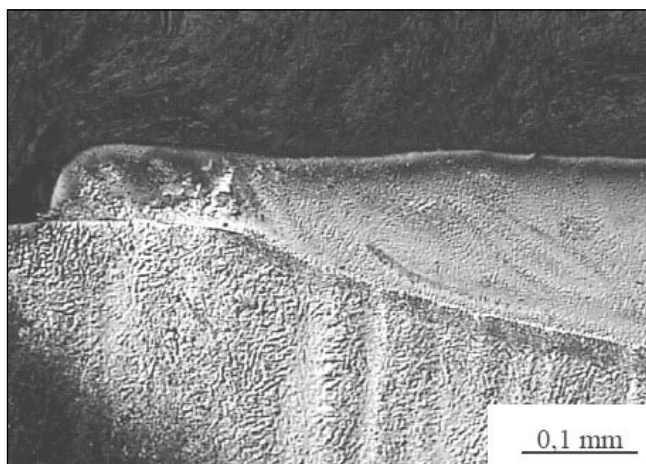
Fig. 2a – Detail of the microstructure of X40CrMoV5-1 steel after laser treatment with parameters: beam power – 1.7 kW, WC layer thickness – 0.11 mm, scan speed 0.5 m/min.

Fig. 2a – Particolare della microstruttura dell'acciaio X40CrMoV5-1 dopo il trattamento mediante laser con i seguenti parametri: potenza del fascio 1.7 kW, spessore dello strato di WC 0.11 mm, velocità di scansione 0.5 m/min.
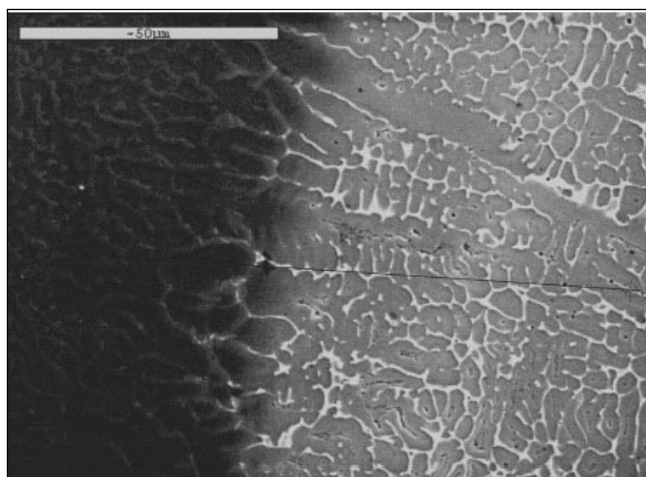

Fig. 2b – SEM microstructure of X40CrMoV5-1 steel after laser treatment with parameters: beam power – 0.9 kW, WC layer thickness – 0.11 mm, scan speed 0.5 m/min.

Fig. 2b – Microstruttura al SEM dell'acciaio X40CrMoV5-1 dopo il trattamento mediante laser con i seguenti parametri: potenza del fascio – 0.9 kW, spessore dello strato di WC 0.11 mm, velocità di scansione 0.5 m/min.

ser radiation, like the light beam, is subjected to the laws of reflection, refraction and absorption, propagating in various media. The absorption coefficient is not constant during the laser treatment and increases significantly when the oxides layer develops on the heated material surface and after attaining the melting temperature by this material [14]. Material transport in the molten material produced by surface tension forces features the main factor deciding forming the alloy layers. The non-uniform material heating due to laser beam impact causes the big surface tension gradient on liquid surface. The force is directed outside from the beam centre, where there is the highest temperature, to its borders, and causes movement of the molten material. Quick crystallization leads to structure diversification in the remelted zone transverse section. Mixing of materials proceeds according to various mechanisms, depending on the employed treatment parameters. Capillary lines are not connected and the remelting structure is relatively homogeneous at low energy values of the laser impact on the material. The capillary lines swirl occurs along

with the laser power increase (Fig. 2), as they begin to link. The remelting bottom is still flat, however slight waviness appears in surface. Employment of the maximum laser power results in obtaining the maximum remelting thickness; however, the remelting bottom gets wavy because of the strong liquid motions. Along with the laser power increase, and the resulting beam impact on the material, increase of the remelting zone depth occurs because of the thermal conduction. Occurrences of the remelted- and heat affected zones have been confirmed in the surface layer of the investigated steel, whose thickness depends on the employed laser treatment parameters. Carbide clusters appear in the central area of the remelted zone, arranged in the characteristic swirls, caused by the convection motion of the material in the liquid state. Size of the crystals depends on the remelting process parameters, and especially on the remelting rate and volume of the remelted material, and also on the non-remelted volume of the heat abstracting material. Solidification rate grows along with shortening of the time period of the laser beam impact on the material and with the increase of the treated material volume, and the obtained crystalline structure is more refined.

The chemical composition of the steel and conditions of its crystallisation affect phase transformations occurring during the colification and cooling processes. Due to the quick crystallisation of the hot-work tool steels after their laser remelting precipitation of the high-temperature ferrite from the liquid solution is observed most often. Next, the peritectic reaction occurs, which leads to origination of austenite. Concentrations of carbide and alloying elements, that – depending on their portion - may precipitate directly from liquid, demonstrating clear segregation at the dendrite boundaries, have a significant effect on austenite origination.

Enrichment of the steel surface layer with tungsten (Fig. 2b, 3) is a consequence of using for alloying the commercially available tungsten carbide powder being a mixture of the WC and W_2C interstitial carbides and pure tungsten (Fig. 4). Tungsten introduced to steel is present in the remelted zone only; however, its concentration grows at the dendrite boundaries, like of other analysed carbide forming elements (Cr, V) (Fig. 4-8). Grouping of the alloying elements was confirmed at dendrite boundaries in the area of the superfine eutectics occurring in the remelted zone due to fluctuation of the chemical composition, especially at the remelting bot-
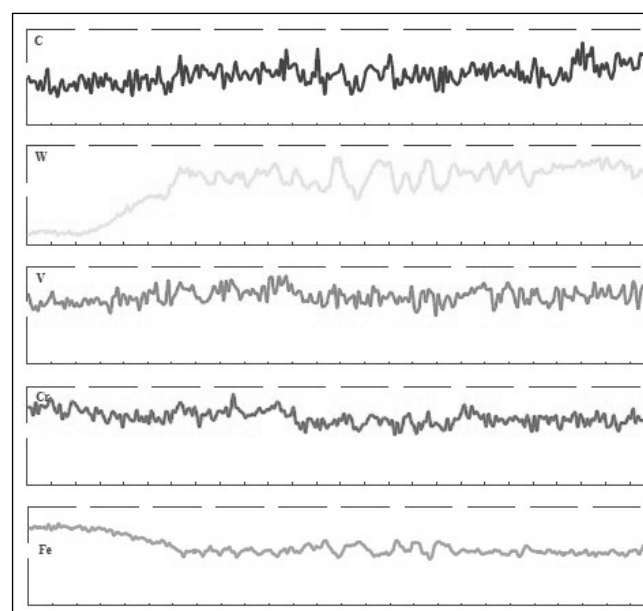

Fig. 3 – Chemical composition through the line shown in Figure 2.

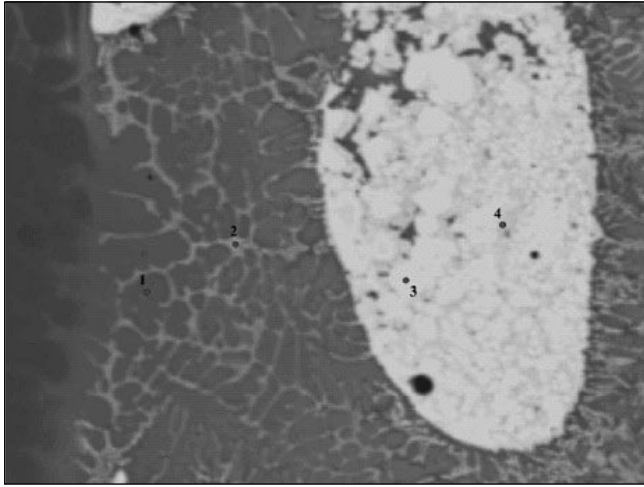*Fig. 3 – Composizione chimica lungo la linea mostrata in Figura 2.*

Fig. 4 – SEM microstructure of X40CrMoV5-1 steel after laser treatment with parameters: beam power – 0.9 kW, WC layer thickness – 0.06 mm, scan speed 0.5 m/min.

Fig. 4 – Microstruttura al SEM dell'acciaio X40CrMoV5-1 dopo il trattamento mediante laser con i seguenti parametri: potenza del fascio – 0.9 kW, WC spessore dello strato di WC 0.06 mm, velocità di scansione 0.5 m/min.
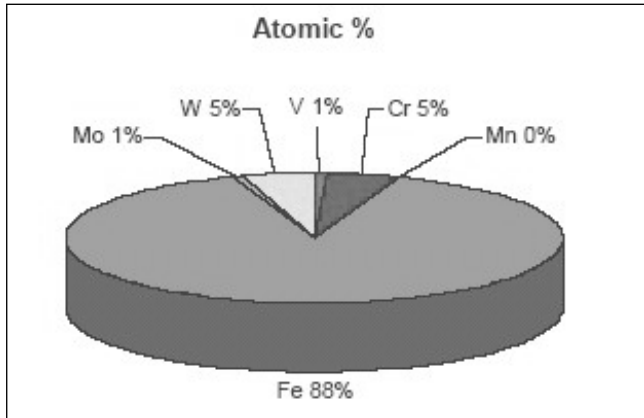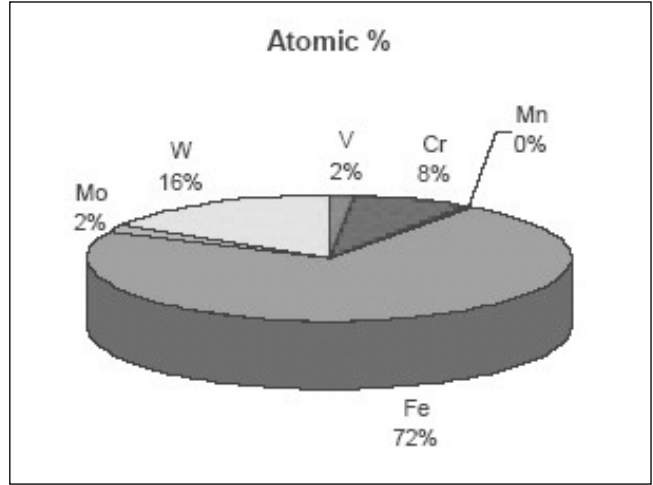

Fig. 6 – Atomic percentage for Point 2 shown in Figure 4.
Fig. 6 – Percentuale atomica nel Punto 2 mostrato in Figura 4.

Fig. 5 – Atomic percentage for Point 1 shown in Figure 4.

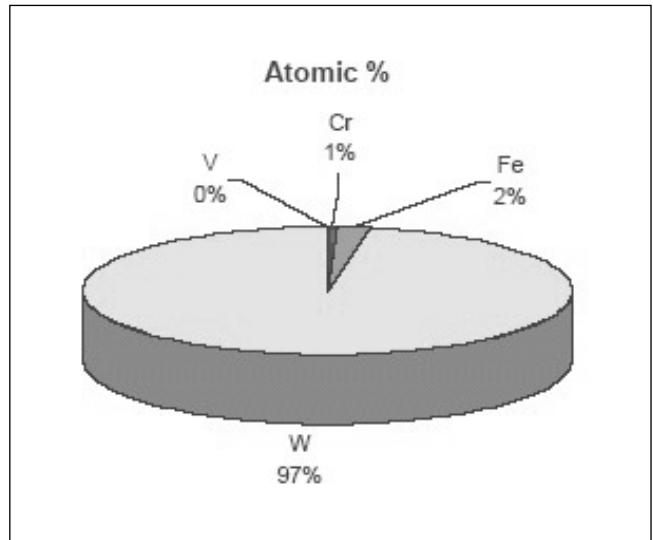Fig. 5 – Percentuale atomica nel Punto 1 mostrato in Figura 4.

Fig. 7 – Atomic percentage for Point 3 shown in Figure 4.

Fig. 7 – Percentuale atomica nel Punto 3 mostrato in Figura 4.

tom. Tungsten carbides and products of its high-temperature decomposition accumulate also at the remelting path edges and in flashes. This mechanism is most surely connected with the difference of the surface tensions of the molten steel and of the alloying material, as well as with the impact of the strong protective gas stream.

The significant cooling rates, occurring during the laser treatment of the surface layer, which at the initial period of the process flow may reach up to 10^{11} °C/s, thanks to the contact of the remelted layer with the non-remelted substrate cause that cooling rate reaches up to 20 m/s. This is the reason for the ultra-fast phase transformations influencing the structural mechanism of forming the surface layers subjected to laser modification. The austenite grain size has an essential effect on the martensitic transformation, and grain boundaries feature the privileged location of the martensitic transformation initiation. Atoms at grain boundaries participate in the martensitic transformation to a less degree because of their weaker bonding with the crystal lattice. Also the crystalline structure defects migrating to the grain boundaries – dislocations – decrease the probability of martensite origination inside the austenite grains. Grain boundaries, except the low-angle ones, feature also obstacles to the martensite lathes' growth, due to the grain crystalline continuity loss (Figs. 9, 10).

Grain size decrease impedes the martensitic transformation flow and leads to increase of the retained austenite portion in steel. Additions of the alloying elements lower the Ms temperature, affecting morphology of the developing martensite. It is most often the lathe martensite in the hotwork tool steels; and several martensite packets separated with the wide-angle boundaries originate within one austenite grain. The martensite lathes in the particular packets are separated with the low-angle- or twin boundaries. Therefore, development of the lathe martensite is dominated by slip shear, therefore the martensite lathes are characteristic of the big dislocation density. Twinned areas occur only locally in the martensite of this type (Fig. 9).

Results of examinations of thin foils on the transmission electron microscope indicate that the remelted zone structure is characteristic of the significant refinement of the martensite with the martensite lathes' length several times smaller than of the ones of the martensite developed during the conventional quenching (Figs. 10). Particles of tungsten carbide and the increased tungsten concentration, forming the superfine eutectics, occur in steel alloyed with tungsten carbide. The increased tungsten concentration in the remelted layer results from the tungsten portion in the alloying powder, tungsten carbide melting temperature, and its solubility in the solid solution.
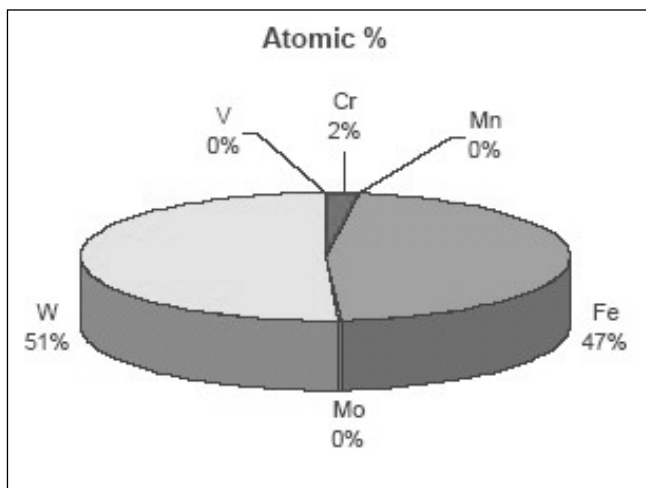
Fig. 8 – Atomic percentage for Point 4 shown in Figure 4.

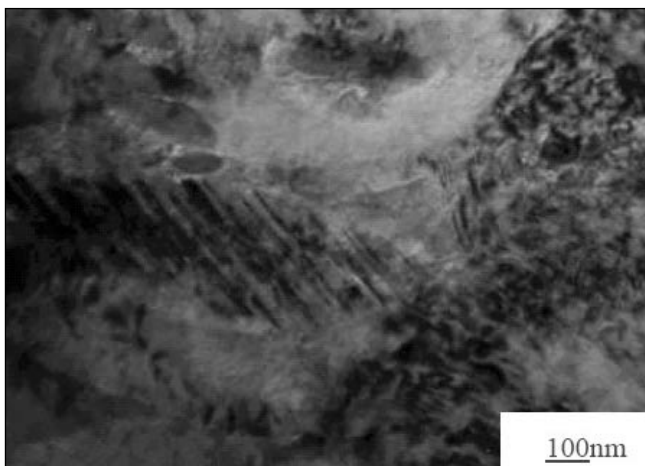Fig. 8 – Percentuale atomica nel Punto 4 mostrato in Figura 4.

Fig. 9 – Thin foil structure from the X40CrMoV5-1 hot-work tool steel after standard heat treatment.

Fig. 9 – Struttura su lamina sottile dell'acciaio da utensile per lavorazione a caldo X40CrMoV5-1 dopo trattamento termico standard.

SUMMARY

It was found out, basing on the research carried out, that it is feasible to develop the surface layers on the X40CrMoV5-1 hot-work tool steel by remelting and alloying with the tungsten carbide using the high power diode laser (HPDL). The structure of material solidifying after laser remelting is characteristic of the diversified morphology connected with the repeated changes of the crystals' growth direction, from the small dendrites, whose principal axes are oriented in accordance with the heat removal directions at the boundary between the solid and liquid phases, clusters of carbides arranged in accordance with the swirls caused by the metallic liquid convection motion, and partially non-remelted WC conglomerates as the alloying material in the central area of the remelted zone, to the fine equiaxial grains in the subsurface zone. The very fast heat removal from 100nm 50nm the remelting zone by the material core with the much bigger thermal capacity, decides the martensitic transformation of the austenite originated due to crystallization, and the lathe martensite developed in this process, partially twinned, is characteristic of the significant refinement of the martensite with the martensite lathes' length several times smaller than of the ones of the martensite developed during the conventional quenching. Laser remelting and alloying with the
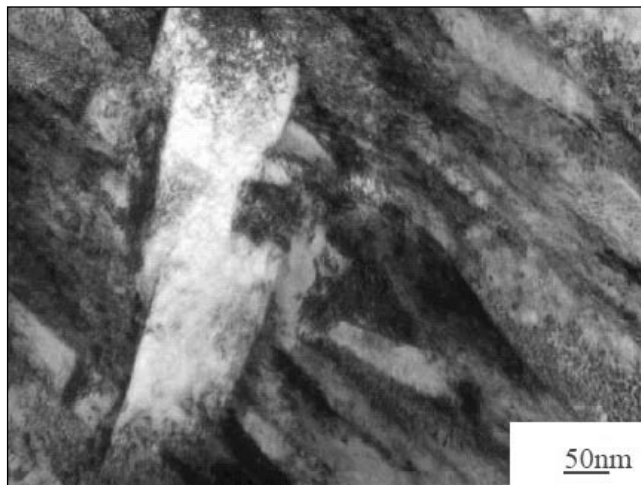

Fig. 10 – Thin foil structure from the X40CrMoV5- 1 steel after alloying with parameters: scanning rate – 0.5 m/min, beam power – 1.5 kW, WC coating thickness – 0.06 mm.

Fig. 10 – Struttura su lamina sottile dell'acciaio X40CrMoV5- 1 dopo alligazione con i seguenti parametri: velocita' di scansione 0.5 m/min, potenza del fascio 1.5 kW, spessore dello strato di WC 0.06 mm.

tungsten carbide results in refinement of the structure in the entire laser power range and in diversification of the grain size in the particular surface layer zones. The grain size in the remelted zone is about twenty times smaller compared to the conventionally heat treated material.

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─────────────── A B S T R A C T ───────────────

EFFETTO DELL'ALLIGAZIONE SUPERFICIALE DI ACCIAI DA UTENSILE PER LAVORAZIONI A CALDO MEDIANTE LASER A DIODI

Parole chiave:
trattamenti superficiali, rivestimenti, tecnologie

L'obiettivo di questo lavoro è lo studio dei cambiamenti microstrutturali dell'acciaio da utensile per lavorazioni a caldo X40CrMoV5-1 durante il trattamento superficiale mediante tecnologia laser. Scopo di questo trattamento sono sia l'indurimento che l' alligazione superficiale dell'acciaio preventivamente rivestito di carburo di tungsteno (WC). E' stato osservato lo sviluppo di uno strato superficiale in cui si distinguono la zona di rifusione, la zona termicamente alterata e la zona di transizione. Nella zona di rifusione e alligata si è evidenziata la struttura dendritica a grano fine, con direzione di cristallizzazione determinata da quella di asportazione dinamica del calore dalla zona di influenza del fascio laser. La struttura martensitica a grano fine è responsabile dell'aumento della durezza dello strato alligato.

Sulla base della ricerca effettuata si è stabilito che sull'acciaio X40CrMoV5-1 è possibile sviluppare strati superficiali mediante rifusione e alligazione con carburo di tungsteno per mezzo di laser di potenza elevata a diodi (HPDL). La

struttura del materiale solidificato dopo rifusione mediante laser è caratterizzata da una morfologia differenziata dovuta ai ripetuti cambiamenti della direzione di crescita dei cristalli, morfologia che va da piccole dendriti (i cui assi principali sono orientati secondo le direzioni di rimozione del calore all'interfaccia fra la fase solida e la fase liquida), a cluster di carburi (disposti secondo i vortici causati dal movimento di convezione del metallo liquido), a conglomerati di WC (parzialmente non rifusi), sino a grani fini equiassiali nella zona sotto la superficie.

La rimozione molto veloce del calore dai 50÷100 nm della zona di rifusione, provocata dalla grande capacità termica del materiale base , è responsabile della trasformazione martensitica dell'austenite ; la morfologia della martensite sviluppata in questo processo è caratterizzata dal sensibile riduzione delle dimensioni degli aghi martensitici che si presentano con lunghezza molto inferiore rispetto a quelle della martensite originata durante la tempra convenzionale. La rifusione e alligazione con carburo di tungsteno mediante laser provoca un affinamento della struttura per tutta la gamma di potenza del laser e la diversificazione della dimensione dei grani nelle particolari zone di strato superficiale. La dimensione dei grani nella zona sottoposta a rifusione è circa venti volte inferiore rispetto a quella del materiale trattato termicamente in modo convenzionale.

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