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Improved Laser Surface Hardening of AISI 4130 Low Alloy Steel with Electrophoretically Deposited Carbon Coating

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Abstract

In the present study, the laser surface hardening of AISI 4130 was conducted using a continues wave diode laser with a maximum power of 1600 W. To improve the laser surface hardening process, carbon powder is deposited on the sample surface by electrophoretic method. By conducting laser surface hardening at the same input parameters and same heat input, the quality of the hardening is compared; cross sectional geometry of the hardened area (width, depth and entry angle of hardened), micro-hardness and the ferrite phase percentage of hardened layer. Microstructure evaluation of the laser hardened area was performed using optical microscopy. Results indicate that because of increasing the laser absorption in carbon coated samples, hardened area is larger (deeper and wider) and the value of the hardness is more than laser hardening of bare samples. Microstructure analysis indicates that transformation of the retained austenite into martensite due to the fast quenching, lead to the lower ferrite phase in martensitic structure in laser hardened layer and causes higher microhardness. Observations reveal that depth, width, and maximum hardness of laser hardening carbon coated sample without surface melting are 1.150 mm, 9.980 mm, and 762 Vickers, respectively, while for bare sample these values are 0.957 mm, 9.451 mm, and 707 Vickers, respectively. Comparing the results with the furnace hardening heat treatment show that the laser hardening process especially with carbon coated, is more effective and precise than conventional processes.

Keywords:

Laser surface hardening; Electrophoretic coating; AISI 4130; microhardness; laser absorption.

1. Introduction

To improve the surface characteristics of a metal alloy such as steels, various types of traditional heat treatment processes are commonly used [1]. However, modern methods like laser surface treatment are more precise [2]. Most recently, laser materials processing has been used for various applications such as drilling [3], cutting [4], welding [5] and surface hardening [6]. Laser surface hardening can cause rapid phase transformation of austenite to martensite, leading to increase the surface hardness. Compared to the traditional heat treatment processes, laser surface treatment can greatly prevent the distortion of the pieces due to its controllable heat input [7]. In the laser surface hardening process, a black coating on the piece surface such as carbon coating can increase the absorption of laser beams and therefore improves surface hardening characteristics [8-12]. In this regard, various techniques employ to deposit the carbon particles on the piece surface. Among them, electrophoretic deposition (EPD) is considered as an effective process for fabricating the uniform carbon coating in order to improve the laser surface hardening properties.

Laser heat treatment has been investigated since the 1970s [13]. Hill et al. [13] employed a 3 kW CW CO_2 laser to increase the surface hardness of a tool steel (0.9% carbon and 1.7% magnesium). In this case, the hardened depth and the maximum surface hardness were obtained about 0.25 mm and 65 HRC, respectively. Benedek et al. [14] in 1980s carried out laser surface hardening process on AISI 1045 (carbon steel), Armco iron, AISI 4340 (alloyed steel) and high speed steel M2 using a 1.5 kW CO_2 CW laser. They investigated the effect of laser movement speed and laser power on the surface hardness. In a similar study, Yang et al. [15] studied the surface hardening of Assab DF-2 tool steel using a 1 kW CO_2 laser. They were looking to find a relation between process parameters (e.g. laser power, beam diameter and scanning speed) and geometrical dimensions of hardened layer (e.g. depth and width). Their obtained results showed that the laser power and scanning speed had a significant role on the hardened depth. Furthermore, they found that the beam diameter had no significant effect on the does not hardened depth while it has an important impact on the hardened width. Effects of laser hardening process parameters are investigated on the characteristic of hardened layer; microhardness value, geometrical dimensions, and microstructure analysis, by so many researchers [16-21]. Increases in mechanical properties have been investigated after laser hardening process; fatigue life [22] and residual stress [23]. Surface hardening of S45C medium carbon steel was carried out using a high power CW Nd:YAG laser by Shin et al. [24]. The effect of the process parameters (e.g. laser power, gas shielding pressure and focal position) on the hardness and hardened geometrical dimensions was studied. They found that the hardness and hardened area width using the designed lens were 3 times larger than those using the defocused beam. The hardened layer width and the maximum hardness were 22.3 mm and 780 HV, respectively. Adel [25] applied Nd:Glass laser for laser surface hardening of Ck45 steel cylindrical rod specimens to improve the microstructure for better wear resistance. In 2014 Sun et al. [26] studied on experimental and FEM numerical simulation of laser hardening of 42CrMo cast steel by a 1 kW Nd:YAG laser by comparing a shaped and a Gaussian laser beam. A comparison study is done by Li et al. [27] to investigate effect of 3.5 kW high-power diode laser and 15 kW CO_2 laser on laser surface hardening of AISI 1045 steel. Their results show that diode laser has a better quality and higher hardness with deeper hardened layer in surface hardening than CO_2 laser. The wear and corrosion resistance also improve after laser hardening compared to the base material [28, 29]. By laser surface hardening the wear resistance of hard alloys (VK6, VK8, T5K10, and T15K6) increases 1.2 times [30]. The wear and corrosion behavior of AISI H13 tools steel improved by 6 kW high power diode laser surface hardening [31]. Increases the hardness from 500 Hv to 810 Hv lead to increasing the corrosion resistance in NaCl 3.56% solution from -920mV SCE to -960mV SCE.

EPD is a favorable and simple method for the fabrication of uniform thin films onto conductive substrates. In this coating process, the charged particles in the prepared stable suspension were uniformly deposited on the surface of the oppositely charged electrode. In this regards, aluminum nitrate or magnesium nitrate are abundantly used as charging agent to positively charge carbonaceous particles in the ethanolic suspensions. Moreover, ethanolic solvent is preferred to aqueous solvent due to its wider potential stability window [32, 33].

Notwithstanding several researches on laser surface hardening, in the literature the diode laser surface hardening of AISI 4130 steel on carbon coated samples have not been addressed before. Herein, in order to improve the laser surface hardening carbon black particles were uniformly coated on the surface of AISI 4130 SS via an electrophoretic deposition method from their ethanolic suspension using aluminum nitrate and polyvinylpyrrolidone (PVP) as the charging agent and dispersant, respectively. By conducting laser surface hardening at the same input parameters using a 1600 kW diode laser, the characteristic of the hardening is compared for coated samples and bare samples. Microhardness distribution and geometrical dimensions of hardened zone (depth, width and entry angle of hardened), and the percentage of the ferrite phase in the structure were investigated. The microstructure of hardened studied by an optical microscope (OM). The results of laser hardening process is compared with furnace hardening heat treatment. The schematic of the laser hardening process on carbon coated sample is shown in Figure 1.

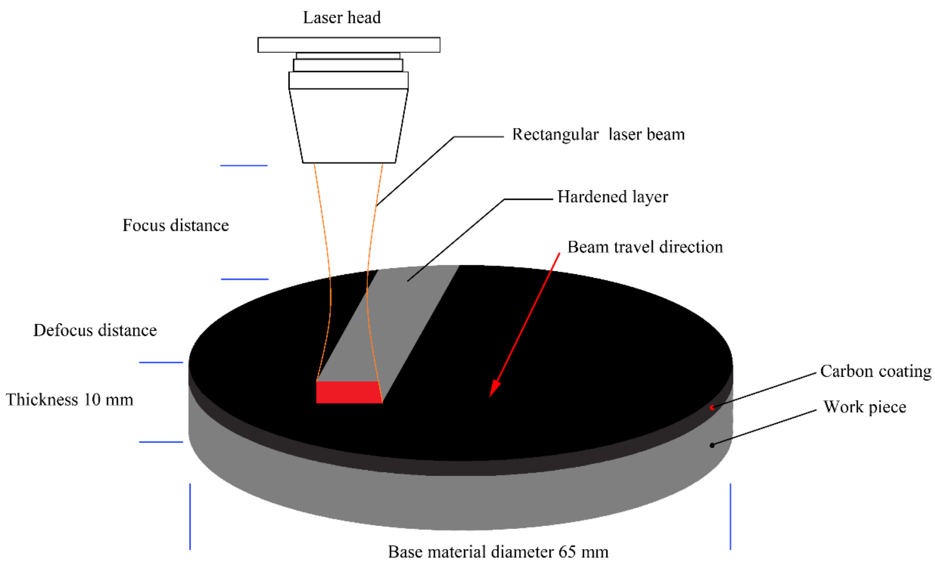


Figure.1. Schematic of the laser surface hardening process

2. Experimental Work

2.1 Material specifications

In this study, AISI 4130 steel specimens were prepared by machining and grinding of its billet with thickness of 10 mm and diameter of 65 mm. The chemical composition (the average of three XRF quantimeter measurements) metals is shown in Table 1.

Table 1 Chemical composition of AISI 4130 low alloy carbon steel (Wt. %)

Element Name	C	Cr	Mo	Si	Mn	Ni	Cu	Al	V	P	S	Fe
Weight percent	0.25	1.01	0.25	0.3	0.87	0.05	0.06	0.024	0.012	0.016	0.03	Balance

2.2 Electrophoretic deposition of carbon black films

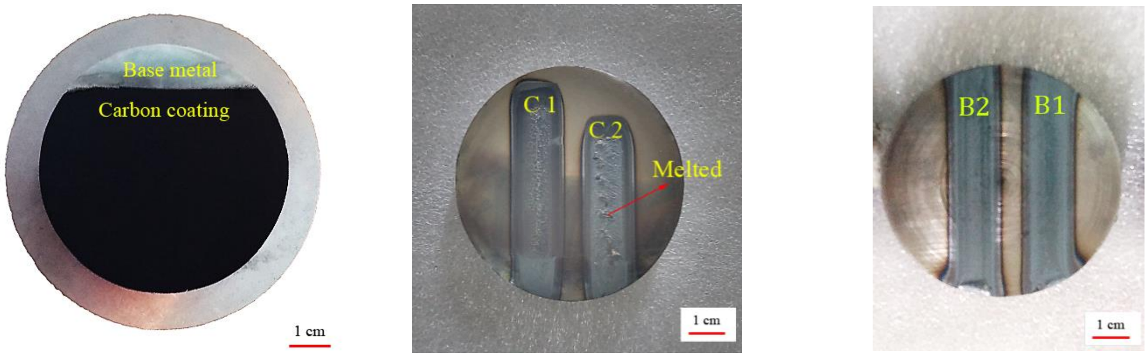
To increase the laser energy absorption carbon is coated on the sample surface by electrophoretic method. Prior to EPD, the surface of SS samples was mechanically polished using abrasive paper (up to 1000 grade) and then repeatedly washed with distilled water and acetone. The suspension for the deposition of carbon black particles was prepared by dispersing 50.0 mg of carbon black particles into 50 mL of ethanol (99.8%, Merck) containing 50.0 mg of Al(NO3)3.6H2O (reagent grade; Merck) and 1.0 mg of PVP (reagent grade; Merck) as the charging and stabilizing agents, respectively. The prepared suspension was ultrasonically dispersed for 60 min before coating process. Cathodic EPD was performed to deposit the carbon film at a constant potential of 30 V for 120 s using a power supply. In this coating process, positive and negative electrodes were spaced 10 mm apart. After coating, the carbon-coated negative substrate was removed from the suspension and then washed with distilled water and finally dried at 60 ºC for 6 h in an oven.

2.3 Laser surface hardening

In order to the surface hardening of the steels, in this study, a diode continuous wave laser with a maximum power of 1600 watts was used. The focal length of this invisible diode laser is 400 mm and its wave length is 808 nm. The focal plane is a rectangle with dimension of 8 mm × 1.5 mm which exactly position on the material surface at 60 mm distance of laser head and material surface. Table 2 illustrates the settings and results of laser surface hardening of AISI 4130. These settings are the optimum results based on the previous research of the authors [34].The same input parameters is used for comparing the laser surface hardening on coated samples and bare samples. Figure 2 shows the image of diode laser surface hardening of AISI 4130 and carbon coated one. As shown in Figure 2-b surface melting is happened in sample #2, coated sample, which is because of increasing the absorption of the laser energy by coating. But in Figure 2-c the melting is not accrued in the same setting.

Table 2. Settings and results of laser surface hardening of AISI 4130 and coated samples

		input parameters			Output responses				
	Samples	S	FPP	P	Maximum hardness (Hv)	Depth (µm)	Width (µm)	Angle ()	Ferrite percent (%)
Coated samples	C1	3.9	64.25	1560	762	1150	9980	12.97	0.37
	C2	4.45	62	1491	813	1427	10716	14.91	0.28
Bare samples	B1	3.9	64.25	1560	707	957	9451	12.51	0.60
	B2	4.45	62	1491	752	1310	9883	13.67	0.41



a) b) c)

Figure 2. a) carbon coated sample b) laser hardened coated samples c) laser hardened baresamples

2.4 Sample analyses

After being hardened using laser treatment, each sample was sectioned perpendicular to the hardened line. To prepare the metallographic samples, the sliced specimens were mounted in phenolic resin, polished up to mirror finish and etched in 2% Nital [35]. The geometrical features of the hardened layer (width, depth and angle) were measured using a BUEHLER MET B7 optical microscope and the images were analyzed by ImageJ software, as shown in Fig. 3. The microstructure was analyzed by optical microscopy (OM). Celemex software was used to measure the percentage of ferrite phase in the microstructure of the hardened layer. Micro-hardness measurements were carried out along a line 50 µm below the top surface on the transversal cross section of the hardened zone using a Micro Hardness V-Test-analog. The micro-hardness was measured with an applied load of 100 g with loading times of 30 s. Each microhardness test was repeated at least three times. Hardness profile was also obtained along the vertical centre line from the top surface to the base metal below the hardened zone. The hardness of the untreated steel was 265 HV.

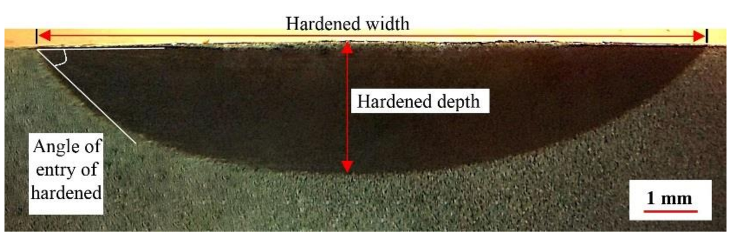


Figure 3. Schematic of geometrical dimensions of hardened zone (width, depth and angle)

3. Results and Discussion

In the present research, the effect of carbon coted on diode laser surface hardening is investigated by comparing with bare samples of AISI 4130 with the same input parameters. To study the surface properties, the geometrical dimensions of the hardened area (depth, width and angle), the micro hardness distribution in depth and width, ferrite percentage, and the micro-structure of the hardened area were analyzed.

3.1 Geometrical dimensions and micro-hardness of the hardened layer

Figure 4 depicts the macro section of four tested specimens and the micro-hardness distribution from the surface to the depth of hardened zone. From Figure 4 it could be easily understand that the coated samples (#C1 & #C2) have higher depth and hardness than the bare samples (#B1 & #B2). This behavior is because of the laser absorption phenomena which increase when the surface of the samples are coated and dark. When the surface of the sample is dark, less percentage of the light reflected and the more part of the light is absorbed by the surface. Material absorptivity by laser radiation is an important parameter effects on the laser material interactions. For opaque materials, the absorptivity (A) can be defined by Equation 1 [36]:

$$A=1-R \quad (1)$$

Where R is the material reflectivity which at normal incidence is defined by Equation 2:

$$R= [(1-n)^{2}+K2]/ [(1+n)^{2}+K^{2}] \quad (2)$$

While parameters n and k are strong functions of wavelength and temperature, the reflectivity (and therefore the absorptivity) of the material is greatly influenced by the wavelength and temperature [36].

The comparison of results of tow experimental settings could be explained by heat input of the laser energy to the material. The heat input presented in Equation (3) is used for better understanding of the process and explaining this phenomenon.

$$\text{Heat input} = \text{Laser power} / \text{Scanning Speed} \quad (3)$$

Thus heat input increases when the laser power increases and the scanning speed decreases. By reducing the scanning speed the interaction time of the laser and material will be enhanced. Then more part of the surface of the material will be heated and the depth of hardened layer increases. Increases in the heat input lead to increases the hardness, while for #B1 and #B2 the heat input are 400 (j/mm) and 335 (j/mm), respectively. In this study the experimental setting presented in Table 2 show different position of focal plane. As mentioned in section 2 while FPP equal to 60mm, the focal plane is exactly positioned on the surface of the sample. Thus from the concept of the divergence of the laser beam after the focal plane, it could be understand that the incident beam area increases while we farther from the focal plane. The incident beam area for FPP=64.25mm and 62 mm is 23.09 and 16.84. The energy beam density is presented in Equation 4:

$$\text{Beam density} = \text{Laser power} / \text{Incident beam area} \quad (4)$$

By increasing the energy beam density the temperature influenced on the sample surface increases. So the beam density calculated by Equation 4 and for sample#B1 and sample#B2 are 67.56 W/mm^2 and 88.54 W/mm^2, respectively.

According to these explanations, although the heat input of sample #B1 is higher than #B2 but because of higher incident beam area which lead to less beam density the total transferred energy to sample #B1 is less than #B2. So sample #B2 has higher hardness, wider, and deeper geometry in hardened zone, see Table 2.

Comparing samples #C1 and #B1 with the same input setting, from Table 2 and Figure 4, show that depth, width, and maximum hardness of sample #C1 are 1.150 mm, 9.980 mm, and 762 Vickers, respectively, while for sample #B1 these values are 0.957 mm, 9.451 mm, and 707 Vickers, respectively. These numbers says a supremacy of using carbon coated. While the more laser energy absorbed from the dark coated surface, the more heat transfer to the material, which leads to the more volume of the material reach to austenite temperature and then by cooling more mass of martensitic field created. In Table 2 it could be seen that the ferrite percentage of sample #C1 is 0.37 and for sample #B1 is 0.60. Having more martensite and less ferrite lead to increases in the hardness.

The comparison of samples #C2 and #B2 with the same input setting is presented in Table 2 and Figure 4. An increase of 1mm occurs in width of hardened zone in #C2 (10.7 mm) in comparison with#B2 (9.8mm). From Figure 2-b it is visible that the surface of sample #C2 is melted while in the same parameter in Figure 2-c the sample #B2 is not melted. As mentioned before, coating increases the laser absorption. Sample #C2 is called laser melting hardening and sample #B2 is laser transformation hardening. In laser melting hardening the quality of the surface of the sample is not acceptable for many industrial application because of bad surface roughness. In laser transformation hardening (#B2) the hardness of the sample increases without any changes in the surface quality just with a transformation in the material phases by heat treatment. Thus, although the hardness of sample #C2 is 813 Hv (61 Vickers more than #B2) and its geometrical dimensions are higher, because of the mentioned reason, sample #B2 is better than #C2.

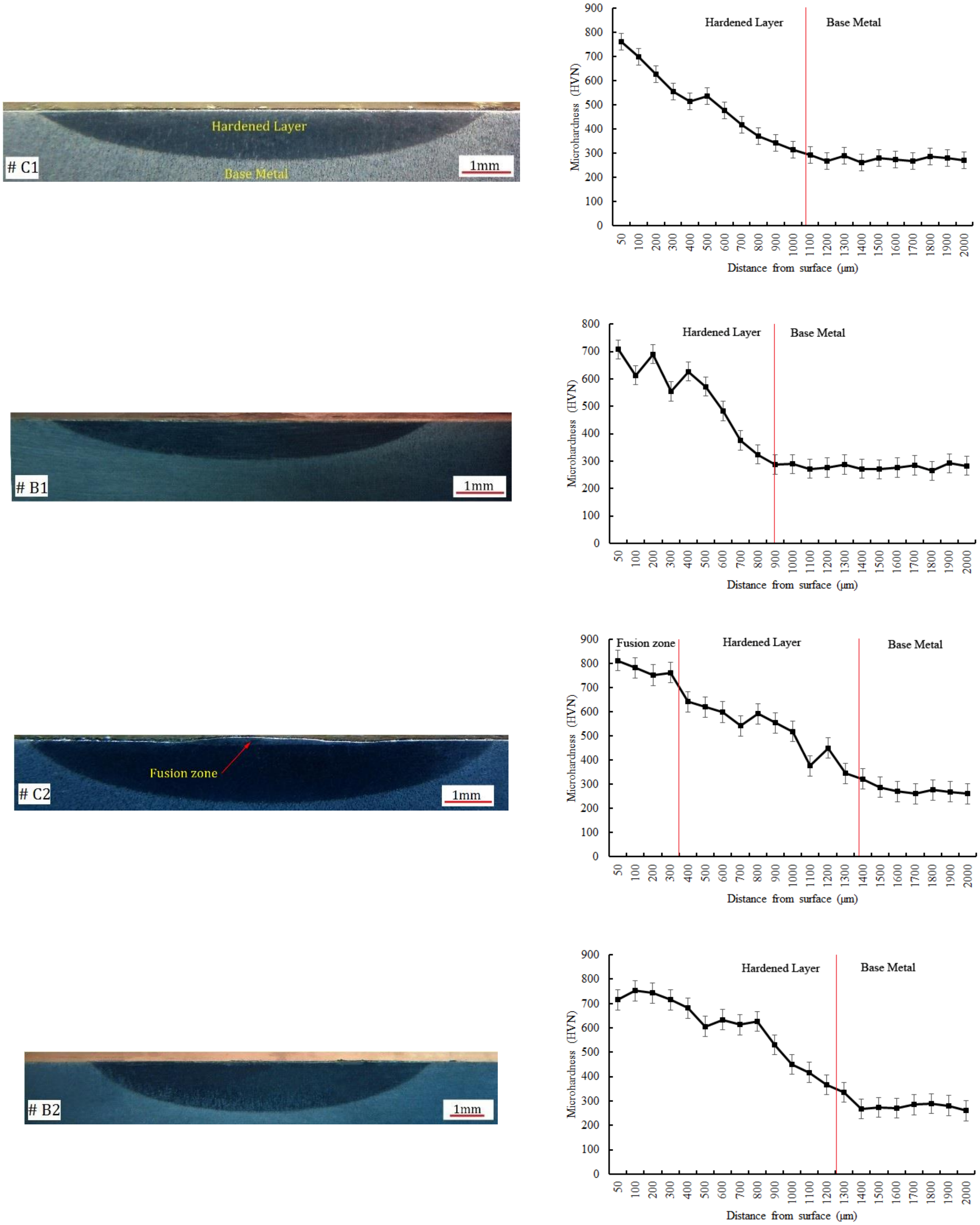


Fig 4. Cross section of laser hardened samples and micro hardness distribution in depth of hardened layer

Figure 5 displays the microhardness distribution in width of the laser hardened layer from the surface to the depth of hardened samples for coated and bare samples. As could be seen in Fig 5 the hardness values of the coated samples (#C1 & #C2) are higher than the hardness of the bare samples (#B1 & #B2). In Equations 1 and 2 it was explained that laser absorption is increased by having a dark carbon coated on the sample surface during laser hardening. Less energy is reflected and more energy is absorbed. Then by having more energy transferred to the material, the temperature is increased and because of metallurgical transformation and martensite formation, the hardness will be increased.

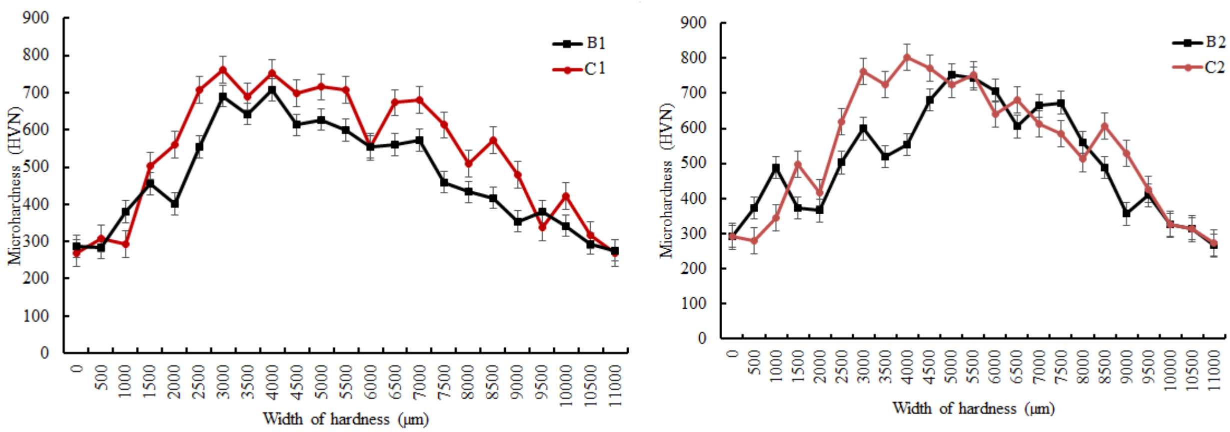


Figure 5. Microhardness profile distribution in width of the laser hardened layer; comparison of coated and bare samples

3.2 Microstructure analysis

Fig. 6 depicts the microstructure of the untreated AISI 4130 material etched in 2% Nital solution (Ethanol 98%, Nitric Acid 2%) which contains ferrite phase distributed in the martensitic field. Compared to the untreated sample, the microstructure of the laser treated samples with and without carbon coating are shown in Fig. 7a-d at various magnifications. As can be observed, the amount of ferrite phase was gradually decreased and the amount of martensite phase was increased after laser processing for both bare and coated samples. Moreover, compared to the bare sample, the carbon coated sample presents a higher level of martensite phase and lower amount of ferrite phase resulted from enhanced laser beam absorption because of positive effect of black coating of carbon. Absorption of laser radiation can be useful as long as it does not lead to surface melting.

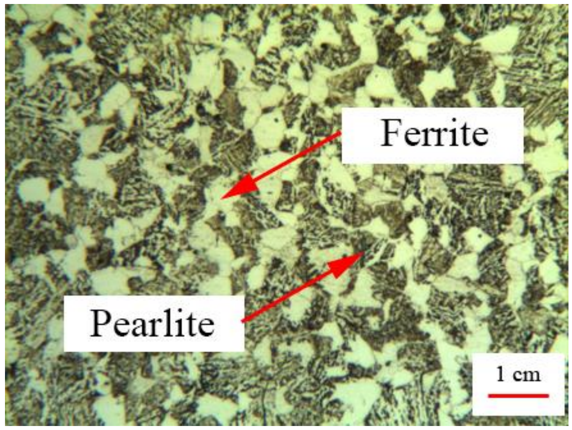


Figure 6. Microstructure of base material AISI 4130

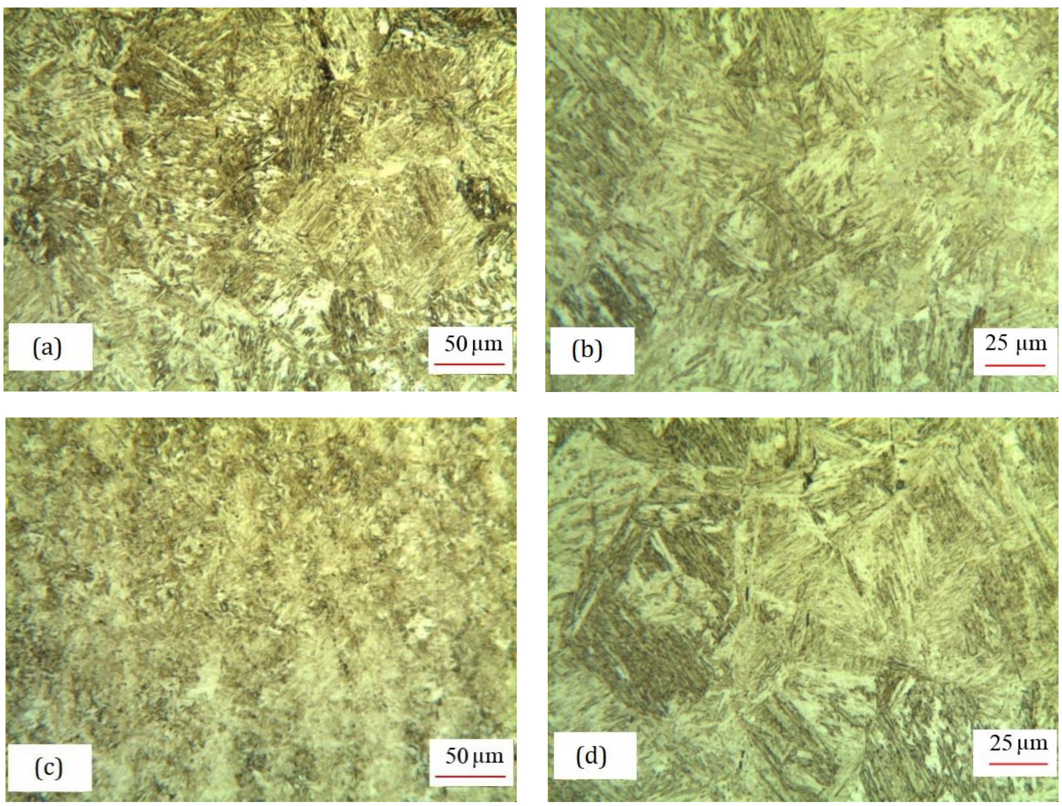


Figure 7) a and b) Nano coat samples (C1)c and d) Bare samples (B1)

For comparing the laser surface hardening method with conventional method, the furnace heat treatment according to cycle presented in Figure 8 was conducted [37]. The prepared cubic samples with dimension of 10mm ×10mm×10mm preheated to 540 ºC for 90 minutes and heated to 899 ºC with the rate of 73 ºC/hour and was kept for 10 minutes [37]. The samples quenched in oil, water and air and the hardness.

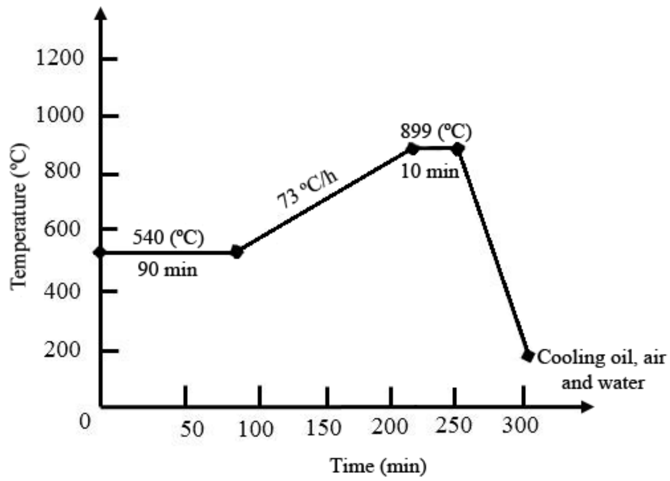


Figure 8. Furnace hardening heat treatment cycle of AISI 4130 used in this research [37]

Table 3 shows the comparison of the hardness results of the furnace hardening heat treatment and the laser surface hardening. Due to the possibility of micro-crack formation in the water, and the lower the hardness in the air, the oil is an ideal quenching method with a hardness of 572 Vickers for AISI 4130. So the hardness value with the diode laser hardening method for bare sample and coated samples (707 & 762 Vickers) are 1.23 and 1.33 times than the furnace hardening heat treatment, respectively.

Table 3 Comparison of furnace hardening heat treatment and laser hardening

Heat treatment cycle	furnace hardening heat treatment	laser hardening
Cooling in oil	572 Vickers	-
Cooling in water	681 Vickers	-
Cooling in air	421 Vickers	-
Bare Sample (#B1 table 2)	-	707 Vickers
Coated Sample (#C1 table 2)	-	762 Vickers

Fig. 9 presents the microstructural observations of the furnace heat treated sample cooled in oil. According to the obtained metallographic images, a higher level of unsolved ferrite phase can be observed in the traditional furnace hardened sample in comparison with the laser hardened sample (Fig. 7). The increased level of the remaining ferrite phase leads to reduce the surface hardness, as can be seen in table 3.

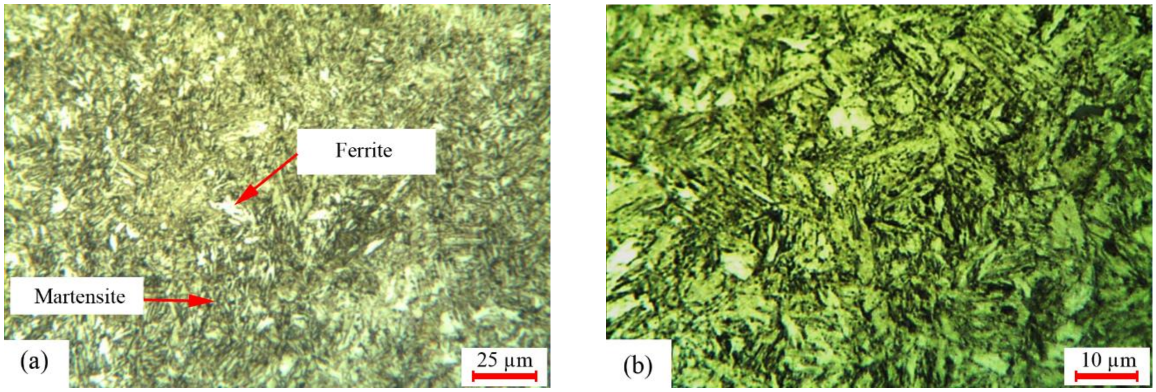


Fig. 9 a) Microstructure of furnace hardened quenched in oil magnitude of 200 b) Microstructure of furnace hardened quenched in oil magnitude of 500

Conclusions:

In this study, diode laser surface hardening of AISI 4130 is improved by carbon coating via electrophoretic deposition technique. According to experimental works, the following results concluded:

1. A uniform layer of carbon was deposited on the surface of AISI 4130 steel specimen via electrophoretic deposition technique using its ethanolic suspension and the effect of this film on the laser surface hardening was investigated in detail.
2. At the same input parameters, higher hardness and larger hardened area is occurred in carbon coated samples than the bare samples due to more laser energy absorption.
3. Electrophoretically deposited carbon coating leads to enhance laser beam absorption and therefore causes to the increased level of martensite phase and higher surface hardness.
4. According to the obtained results for surface hardness and microstructural characteristics, it is found that laser surface hardening process is more effective than conventional furnace hardening process.
5. The maximum hardness value for the laser hardened coated samples is 762 Vickers while this value for the laser hardened coated samples is 707 Vickers and for furnace hardened samples is 572 Vickers. Because there is lower ferrite phase in laser hardened samples compared to furnace hardened samples.

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