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Surface Improvement of Low Carbon Steels (St37) by Duplex Treatments

Yıldız YARALI ÖZBEK*[1]

Abstract

The aim of this work was seen to effect to duplex treatment on steel surface. Firstly, arc spray and secondly pulse plasma treatment were applied to sample surface. The influence of these duplex process and process parameters want to see on surface. Swm Inox 446-TW wire was used in arc spray process. Then the pulse plasma process was applied to surface. The different distances and number of pulse were used as the pulse plasma treatment. After duplex treatments, the samples were characterized by using OM (optical micrograph), scanning electron microscopy (SEM-EDS). X-ray diffractometer (XRD) analyses and microhardness tests were applied. The surface roughness of samples was measured with 3D-roughness device. The microhardness values of treated surfaces are about four times higher than that of un-treated surfaces. After duplex treatments, the improved performance of surface the St 37 steel was presented.

Key words: surface, steel, duplex treatment, arc, pulse plasma

1. INTRODUCTION

The arc thermal spraying has been effective in long-term anticorrosion and wear properties of the steel structures in the aggressive conditions [1-4]. Especially, the wear failure of the samples begins mostly on the surface or subsurface. The coating to the surfaces of a component can effectively decrease wear. So, the component's service life of sample was improve [2]. The arc spraying yields production of the coatings with good bonding strength and a low degree of porosity. Arc Spray is the most economical and efficient of all thermal spray coating systems [4].

The wire are melted. The melted metal is atomized into molten micro-droplets using compressed air, and the droplets are sprayed onto a surface. Arc Spray has two conductive wires: one positive and the other negative. These energized wires are raised through a feeder into a gun head. It is at the gun head that the wires compensated and arc against each other, thus formed molten material [5,7].

The dry compressed air transferred to the arc zone, atomizing the molten material into small droplets while also pushing them toward the prepared part. The droplets hit to the work piece or part, and flatten out and make splats [2]. The splats connect one on top of another to create an extremely intense mechanical bond. An air stream the back of the gun atomizes and propels the molten particles towards a

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prepared surface. The regulable parameters including voltage and amperage arc temperatures can reach 5000° C, enough to melt any material, simultaneously the part surface continue cool. All of the entry energy is used to melt the spray materials. So, the arc spray is considered as an energy efficient process [3]. Only the porosities are problem in this system.

the pulse plasma process is another technique used to devolope the surface properties of the steel surface [6]. The pulse treatment distributed to surface, the near surface layer of the target pass on a rapid melt and solidification with heating and cooling rates from 10^7 to 10^{10} K/s. These rates can supported mixing, rapid diffusion, and the formation of amorphous surface on sample. Therefore, the mechanical properties of material surfaces can be increased [8-10].

The surfaces of the samples were subjected to pulse detonation process accompanied under a plasma atmosphere containing alloying elements dissolved from the metal electrode rod in interaction with the constituents of propane and nitrogen gases used for the process. The tungsten was chosen as consumable electrode in this work. This process provided the workpiece surfaces with alloying of plasma components and hardening of surfaces [1,4].

The Duplex process was applied to sample surface to develop the surface properties [8]. The purpose of the present paper is to investigate the effect of the duplex coatings prepared with an arc spray process and pulse plasma technique. The aim of the study is to decrease the amount of the pores after duplex trearment. We want to see the effect of process and parameters. There were some porosite on surface coated by arc spray process. The pulse plasma process may solve porosite problem.

The optic and SEM micrograph were taken from cross-section of samples. XRD, EDS analyses were applied. The microhardness were taken from surface by microhardness apparatus. The surface roughness was measure 3D- roughness device.

2. EXPERIMENTAL PROCEDURE

St37 steel was chosen as the substrate material for the surface modification, because it is cost-effective and commonly used in industrial applications. The chemical composition of the St37 steel was given in Table 1. The diameter of the cylindrical samples was 22 mm, and their thickness was10 mm. They were machined with a CNC lathe. The machined samples were not subjected to any heat treatment prior to duplex treatment.

The sand blasting process was applied to sample surface. The SWM INOX 446-TW wire of about 2 mm in diameter was used in the arc spraying material. The chemical composition of arc wire used in arc spray process was given in Table 2.

The process parameters that were applied to the machined samples were presented in Table 3.

After the arc spray process, all samples were exposed to pulse plasma treatment. The arc spray process parameters were given in Table 3.

Table 1
The chemical spectrum of the ST 37 steel

	(%wt.)			
Steel	C	P	S	N
St37	≤ 0,17	0,035	0,04	0,009

Table 2
The chemical composition of the arc wire

Wire (%wt)	%C	%Cr	%Mn	%Si
SWM INOX 446-TW	0.3	13.0	1.0	1.0

Table 3
Parameters of arc spraying

Technological parameters	Value of parameters
Arc voltage (V)	34
Arc current (A)	150
Atomizing gas pressure (MPa)	0.6 ± 0.1
Spraying distance (mm)	150 ± 20
Feed voltage (V)	100

2.1 Pulse plasma system

Fig.1.shows the schematic of the pulse plasma process. The pulse plasmatron formed in a detonation chamber where the fuel gas mixture is occurred and its detonation combustion is initiated, a central electrode-anode, a conical electrode-cathode, inter-electrode gap

(4), a consumable electrode (*5)* and a power supply (*6)* [3].

At the beginning of the detonation the ionized combustion products were fed from detonation chamber (*1)* to the inter-electrode gap (*4)* in order to fulfil the electric circuit. This led to formation of conducting layer (*7)* formed by the combustion products under the effect of gas-dynamic and electromagnetic forces. The consumable metal rod (*5)* was fixed in a position along the axis of central electrode. The end of the rod was evaporated during heating and enabled alloying elements to be introduced into the plasma jet. After the samples were ejected from the plasmatron, plasma jet (*8)* had broken the circuit between the anode electrode and the cathode work piece (*9*). The current flowing through the jet the plasma was heated due to Joulean heat [1,3]. The pulse plasma process parameters were given in Table 4. The tungsten electrode was used for pulse plasma treatment

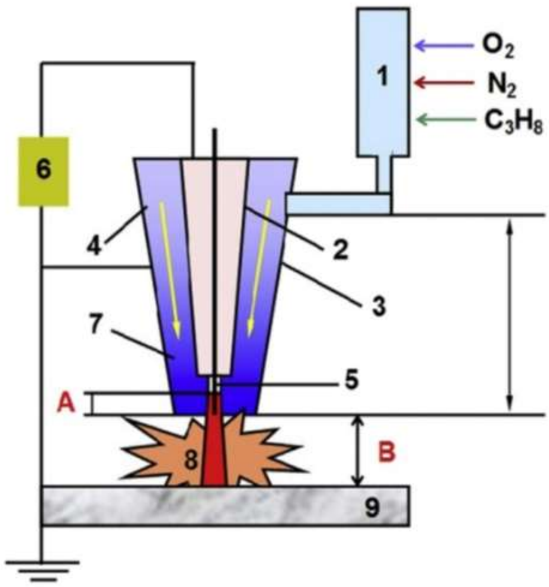


Figure 1 Schematic presentation of the pulsed-plasma modification system

The microstructure of the coatings was examined using an optical and scanning electron microscope (Leica and Jeol) equipped with energy dispersive spectroscopy. The phase composition of the duplex layer was determined by means of X-ray diffraction (XRD; Rigaku 6600) with Cu-Kα radiation (λ = 1.54 Å) operated at 40 kV, 40 mA and with steps of 0.02°. The microhardness test was carried out on the cross-section of the coating (Leica-1000TC) under a load of 10gr. The hardness value was calculated from the average of 5 measurements for each sample to ensure the data repeatability. The roughness of coated surface was measured with 3D profilometer.

Table 4
The process parameters of pulse plasma treatment

Sample No	*Nozzle distance (mm)*	*Number of pulse*	*Electrode*	*Battery capacity (mf)*
1	*50*	*4*	*W*	*800*
2	*50*	*8*	*W*	*800*
3	*50*	*12*	*W*	*800*
4	*60*	*4*	*W*	*800*
5	*60*	*8*	*W*	*800*
6	*60*	*12*	*W*	*800*
7	*70*	*4*	*W*	*800*
8	*70*	*8*	*W*	*800*
9	*70*	*12*	*W*	*800*
10	*80*	*4*	*W*	*800*
11	*80*	*8*	*W*	*800*
12	*80*	*12*	*W*	*800*

Nozzle distance (mm); distance of between sample-nozzle
Pulse number: number of shots (pulse)
Consumable electrode: tungsten

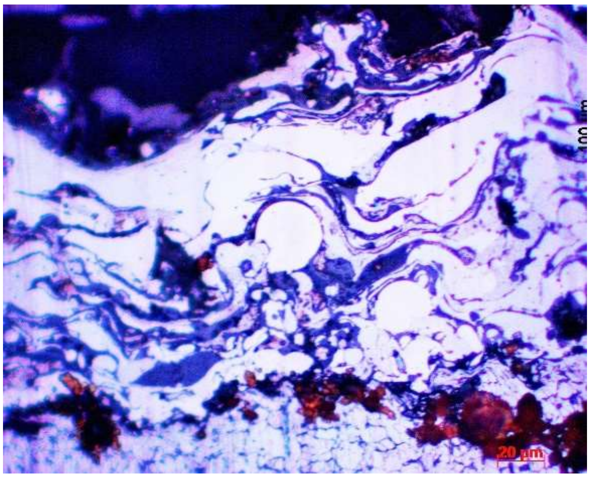
3. Results and Discussion

The optic micrograph of samples 1, 2 and 3 were shown in Fig.2. The cross-sectional microstructure images revealed that the as-sprayed coatings were densely deposited into a lamellar structure with porosity. The structure was homogeneous. The nature of the arc spray has been created some pores The interfacial defect was shown at 2–4%. High temperatures and high velocities of the unmelted particle usually form the important droplet deformation on impact at a sample surface [6]. The formation of pores is closely associated with large unmelted particles. So, a relatively broad range of microhardness was obtained due to the rough surface morphology resulting from pore defects. But we have applied pulse plasma process in addition to arc spray. So the amount of porosity decreased after pulse plasma process [5]. We can see the three phases in optic micrograph. The layer was composed of : the light gray metal matrix, medium gray metal oxides, and black void spaces [3,6].

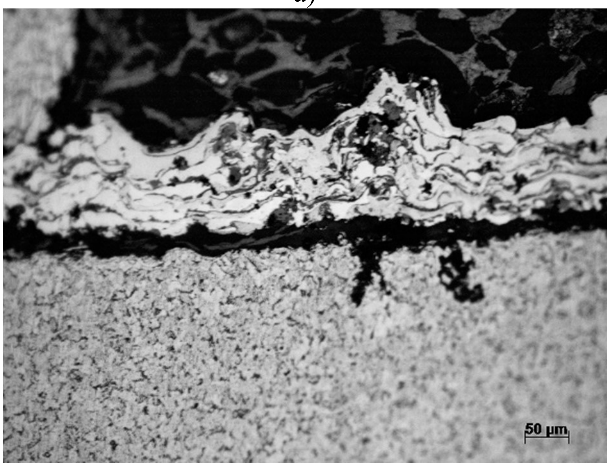
The solidified droplets establish rapidly, as a continuous stream of completely melted particles' impact to form continuous solidified layers. However, the sprayed particles from the nozzle actually reach the substrate in fully melted and semi-melted conditions because of the use of wires as a feedstock material produces different sized particles by the nonuniform heating. When the particles are not fully melted or do not have enough high rate velocity until the collision with the substrate, the particles cannot fully fill the void in the coating and pores are formed.

The SEM micrograph of the all samples were seen in Fig.3. The duplex coating structure was shown from the cross-section. Pulse plasma layer was seen on top of the coated layer (Fig.3a-b-c). The very small pores were seen in the coated layer. The thickness of layer modificated by pulse plasma is thinner than the layer coated by arc spray plasma.

The amount of the oxides (thin grey lamella) in the coating, which were identified by EDS. This indicates that the grey areas in Fig.3a were oxides of Fe or Cr. This was consistent with the XRD results and the oxygen content (analyzed by Oxygen, Nitrogen Analyzer), suggesting the severe oxidation of the coating during the spraying process.



a)



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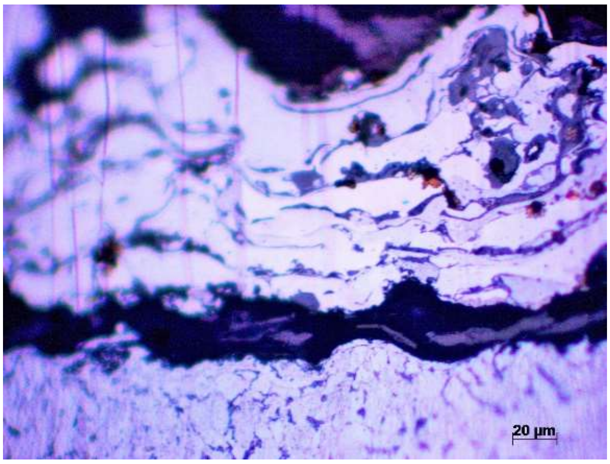
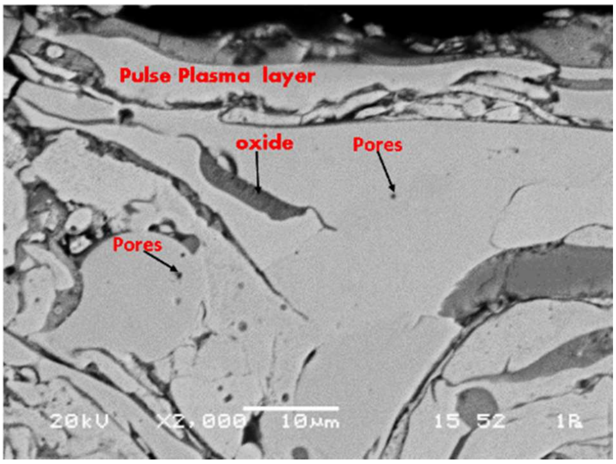
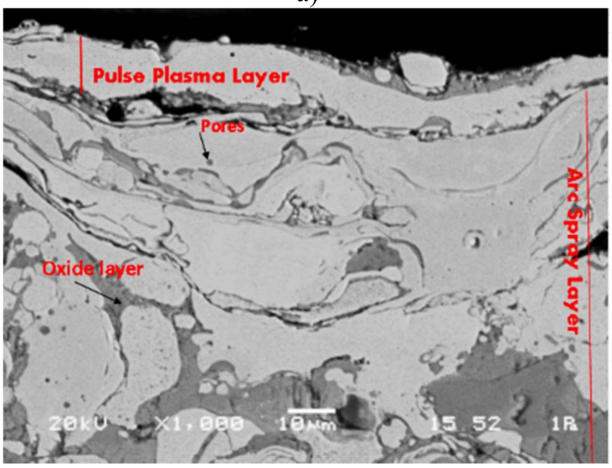


c)

Figure 2 The optic micrograph a) sample 1, b)sample 2, c) sample3



a)



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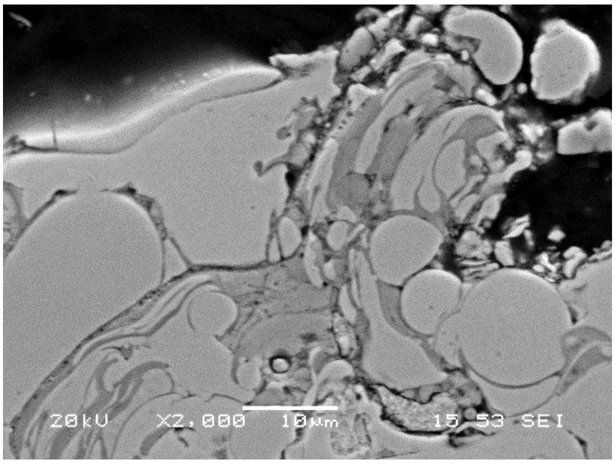

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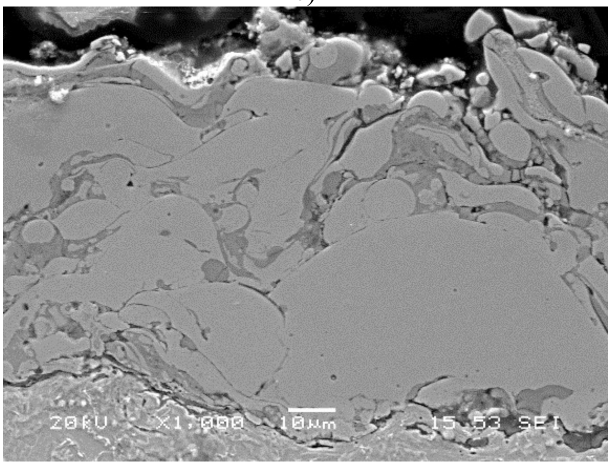

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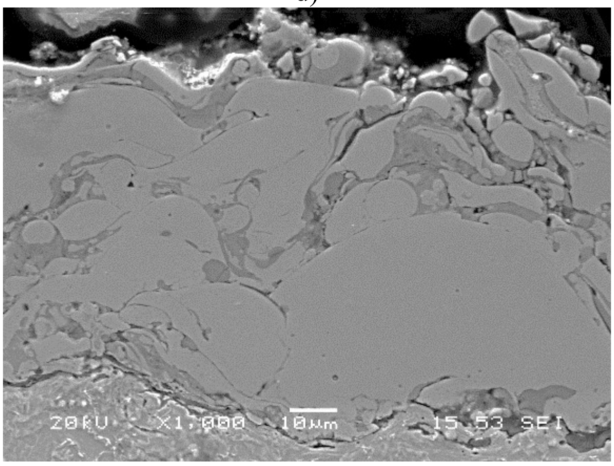
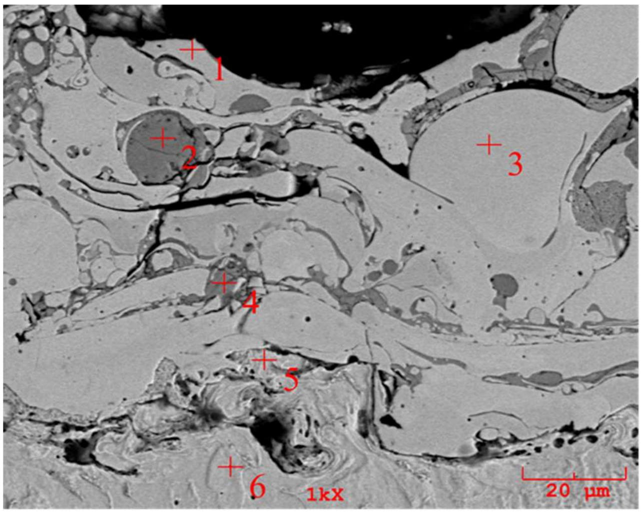


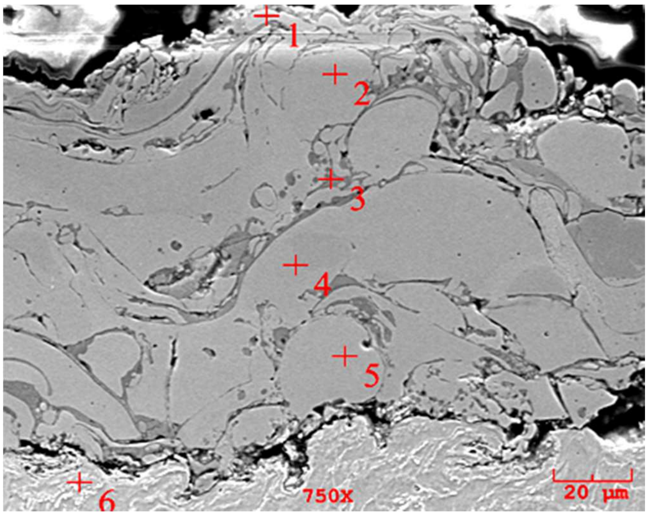
e)

Figure 3 The SEM micrograph of samples a) sample 2, b) sample 3, c)sample 4, d)sample 5, e)sample6



a)

%Wt.	1	2	3	4	5	6
O	5.524	32.769	4.335	27.632	0.612	0
Cr	27.218	12.213	32.983	44.370	1.771	0.240
Fe	67.258	55.018	62.682	27.998	97.617	99.760



b)

%Wt	1	2	3	4	5	6
N	2.093	0.459	0.680	1.214	0.482	0
O	5.916	0	30.307	6.200	0	0
Cr	40.402	27.085	36.724	48.284	29.086	0.388
Fe	48.969	71.258	30.597	43.013	68.459	99.028
W	2.620	1.198	1.693	0.983	1.972	0

Figure 4 The EDS analyses of sample a) arc spray,b)arc spray+pulse plasma proces (duplex treatment)

The Fig.4a shows the EDS analyses applied sample of arc spray and Fig.4b shows sample of duplex (arc spray+pulse plasma) process. The duplex treatment was changing the structure of the coating layer. The nitride and tungsten were seen on the surface after duplex treatment (Fig.4b). The chromium wire was used duration arc spray. The tungsten consumable electrode and nitride gases were used in pulse plasma process. So, these elements have emerged with the duplex process [5].

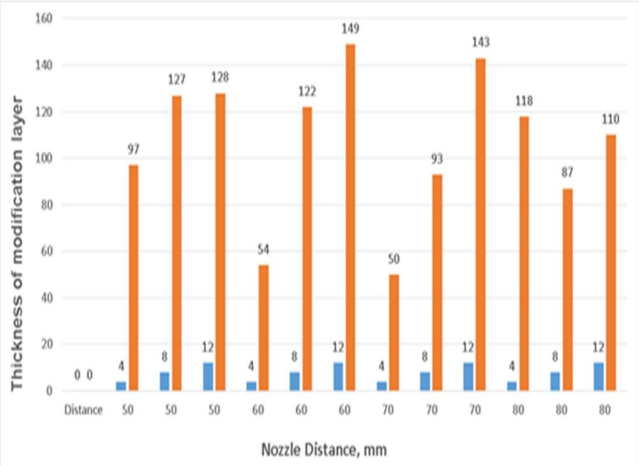
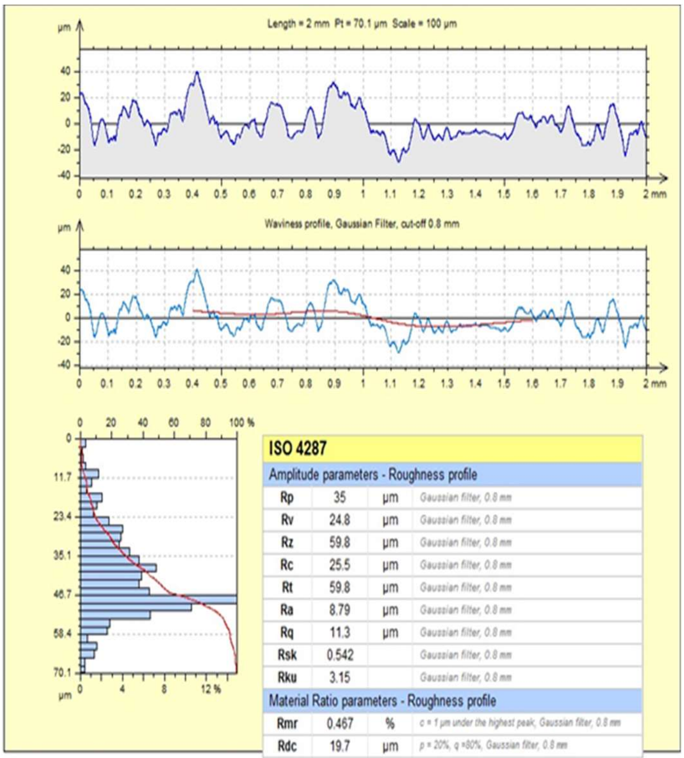


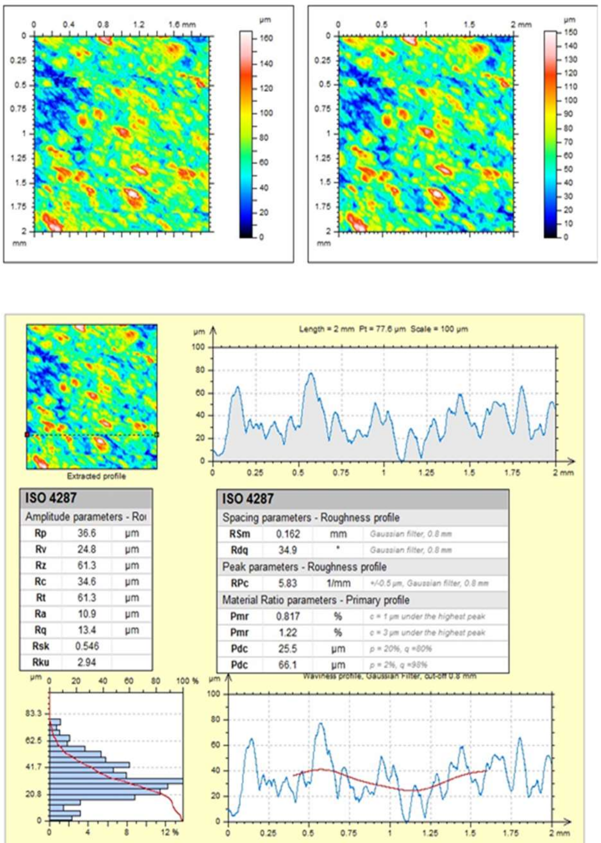
Figure 5 The changing of thickness of coating layer to pulse plasma parameters

After the pulse plasma process, the thickness of then coating layer was measured. The process parameters were effected the thickness of the coating layer (Fig.5). With the increase of the number of pulse, the thickness of coating layer was increased. The nozzle distance was affected to thickness of coating. The focusing problem was occurred in system, ionization gases was randomly dispersed to surface. So the thickness of the modified layer was not compatible with process parameters. However manual optimization in the pulse plasma system could be formed inhomogeneities to surface properties.

The roughness is very changing by arc spray (Fig.6a) and then pulse plasma process applied to surface (Fig.6b). The Ra value was changed from 8.79 to 10.9 (The Ra; arithmetic average deviation). The roughness was increased after duplex treatment. Because of the heat is high. The heat was led to the melting of some areas at the surface. However, changing the heat of the surface was increased the roughness [7].



a)

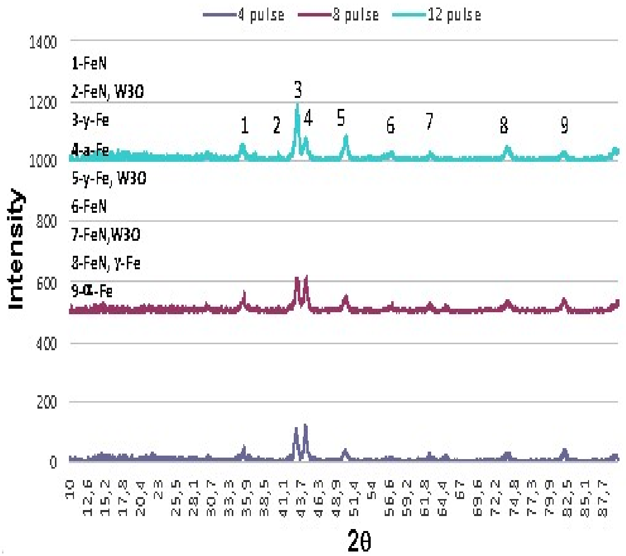


b)

Figure 6 The surface roughness 3D a) arc spray, b) duplex process (arc spray +pulse plasma)

XRD pattern of the samples after duplex treatment is shown in Fig. 7(a-b). substantial phase of the coating is the crystalline phase of α-Fe. The new hard phases were obtained after duplex treatment. Before duplex treatment, there was only α-Fe phase in steel structure, but after duplex treatment new phases such as FeN, W_3O, γ-Fe were formed [8-16].

The coating layer was composed of the amorphous and crystalline structure. The Fig.7b show that the crystalline phase was established in the amorphous matrix. The formation of an amorphous alloy structure requires the high cooling rates. During pulse plasma process, the cooling rate is 10^5 K/s, which is suitable for forming an amorphous phase. W (consumable electrode) can develop the glass forming ability of the coating as a consequence of their additive to the large atom and large negative heat mixing among the constituent elements [7,9,14].



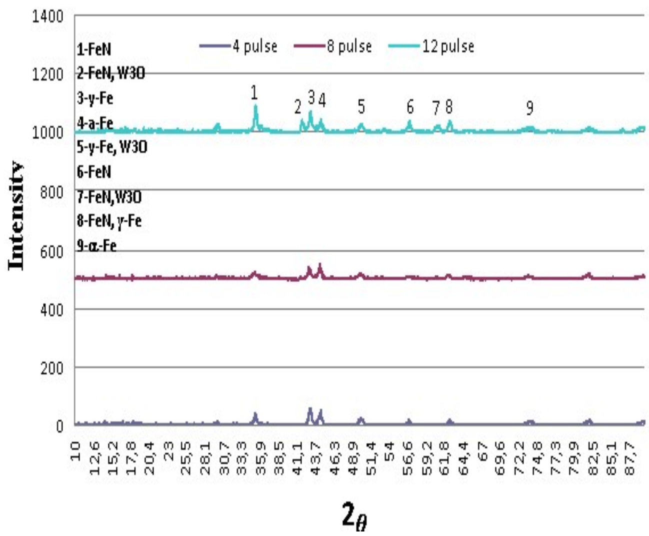


Figure 7 The XRD analyses of sample a) sample 4,5 and 6, b)sample 10,11 and 12

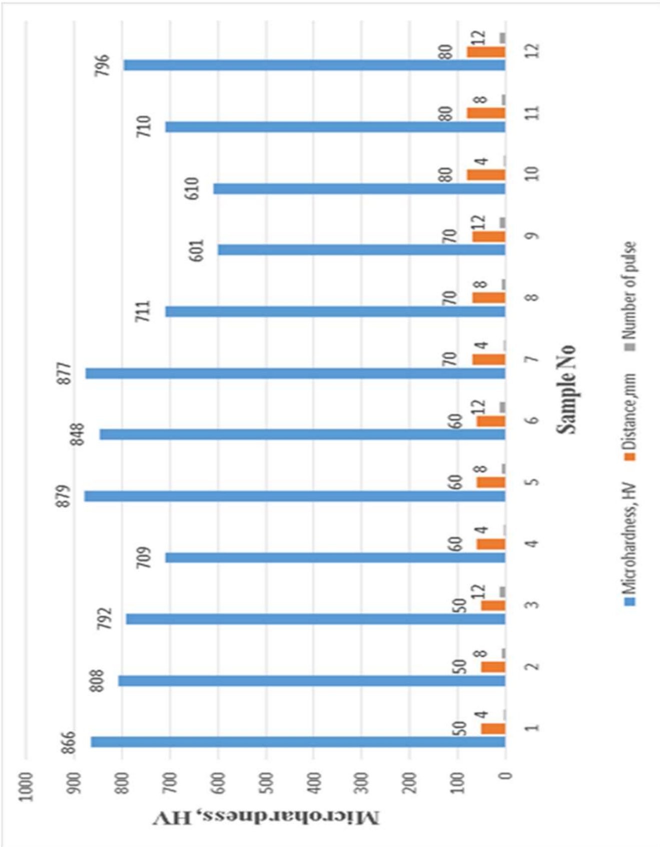


Figure 8 The microhardness values of samples

Fig.8 shows the change of the hardness values after duplex treatment. The hardness of the substrate metal was measured as 180 HV. After the arc spray the hardness values increased up to 400-500HV. The microhardness values were changed with pulse plasma process. The parameters of pulse plasma treatment affected the values of the surface hardness. It can be observed that the hardness values of surface decrease by increasing the spraying distance, presenting maximum values of 879 HV at the spraying distance of 60 mm. When the number of pulse increased, the hardness was increased [13-16]. The amount of ionization gases was increased by increasing the number of pulse. The hardness of modified samples gradually decreases due to the alloy concentration near the core, resulting in a diffused case–core interface. The high cooling rate and solidification of surface melting material could produce very fine microstructure. The microstructure of treated surface will become finer with increasing the number of pulses [16,11].

The surface layer consists of a composed of sub-grains with many defects, such as dislocations and grain boundaries that can be formed using the pulse plasma method. The nanostructured surface layer of the St37 steel significantly promotes the nitrogen diffusion process when many pulses are used, resulting in the formation of new hard phases [14,15,17,18]. The new hard phases were increased the surface hardness [15,16]. The nozzle distance was effected the microhardness values.

In addition, the hard phases and new structure improved the surface hardness [5,12]. Not only the St37 steel but also M2 steel hardness values were increased after the pulse plasma treatment [17].

4.CONCLUSION

In this work, the effect of duplex process on the hardness and microstructure of the samples was studied. Two step process namely the arc spray and then the pulse plasma treatment were applied to surface. The duplex treatment was improved to sample surface. The gradient coating layer was formed on surface layer after duplex treatment.

1) The layer was composed of (i)light gray metal matrix, (ii) medium gray metal oxides, and (iii) black void spaces in arc spray treatment. The white layer occurred on the top layer after pulse plasma treatment.
2) The duplex treatment was increased the surface roughness. The arc spray was caused porosity and low density, but the pulse plasma was covered porosities. The porosity problem was overcome by duplex treatment.
3) The arc spray was caused the partial melting of the surface. The pulse plasma covered the melted area.
4) In the pulse plasma process, rapid heating-cooling and diffusion lead to a change in the surface structure. This new structure improved the mechanical properties of the steel.
5) The consumable electrode (W) causes the ionization of Tungsten (W) and Nitrogen (N) atoms. These atoms are doped into the surface by means of diffusion mechanism.
6) The process parameters, such as the nozzle distance and the number of pulses, were need to be adjusted to improve and change the mechanical properties of the surface. The thickness of the modified layer increased with increasing number of pulses at the optimum nozzle distance.
7) The surface process for the St37 steel produced a surface layer with more favourable properties. New phases formed on the modified surface due to the pulse plasma treatment. tungsten and nitride-rich phases were formed: FeN, W_3O, and γ-Fe. These phases play a key role in developing the increased surface hardness.
8) The duplex treatment was contributed to the mechanical properties. The hardness values of the specimens that were exposed to the treatment were 4 to 6 times greater than the untreated specimens. When only arc spray was applied, the hardness was increased 3 times, whereas the duplex treatment (arc spray+pulse plasma) yields increased hardness up to 4-6 times more than that of the bare steel substrate.

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The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the author.

The Declaration of Ethics Committee Approval

This work does not require ethics committee permission or any special permission.

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The author of the paper declares that she complies with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that she does not make any falsification on the data collected. In addition, she declares that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.”

REFERENCES

[1] H. Tian, C. Wang, M. Guo, Z. Tang, “Tribological behaviour of a self-lubricated GO/WC–12Co thermal spray coating”, Surface Engineering, vol. 353, pp 237-246, 2018.

[2] W. Tillmann, L. Hagen, D. Kokalj, “Embedment of eutectic tungsten carbides in arc sprayed steel”Surface and Coatings Technology, vol. 331, pp. 153-162, 2017.

[3] S.Salavati, T.W.Coyle, J.Mostaghimi, “Twin wire arc spray process modification for production of porous metallic coatings”, vol. 286, pp. 1-396, 2016.

[4] B. Podgornik, F. Majdic, V. Leskovsek, J. Vizintin, “Improving tribological properties of tool steels through combination of deep-cryogenic treatment and plasma nitriding”, Wear, vol. 288, pp. 88–93, 2012.

[5] Y.Y.Özbek, M.Durman, H.Akbulut,“Wear Behaviour of Steel Modıfied by Pulse Plasma Technıque”, Tribology Transaction, vol. 52, pp. 213–222, 2009.

[6] http://www.thermioninc.com/

[7] http://www.mbicoatings.com

[8] X. Zhang, Z. Wang, J. Lin, Z. Zhou, “A study on high temperature oxidation behavior of high-velocity arc sprayed Fe-based coatings” Surface and Coatings Technology, vol. 283, pp. 255-261, 2015.

[9] Y.Y.Özbek, H.Akbulut, “M.Durman, Surface properties of M2 steel treated by pulse plasma technique”, Vacuum, vol.122, pp. 90-95,2015.

[10] J. Piekoszewski, Z.Werner, W.Szymczyk, “Application of high intensity pulsed ion and plasma beams in modification of materials”, Vacuum, vol. 63, pp. 475-481, 2001.

[11] V.V. Uglov, V.M. Anishchik, N.N. Cherenda, Y.V.Sveshnikov, V.M. Astashynski, E.A. Kostyukevich, A.M. Kuzmitski, V.V. Askerko, “The formation of a tungsten containing surface layer in a carbon steel by compression plasma flow”,Surface & Coatings Technology, vol. 202, pp. 2439–2442, 2008.

[12] M.F. Yana, Y.Q. Wua, R.L. Li, “Grain and grain boundary characters in surface layer of untreated and plasma nitrocarburized 18Ni maraging steel with nanocrystalline structure”, Applied Surface Science, 273, pp. 520–526, 2013.

[13] B. Podgornik, S. Hogmark, O. Sandberg, V. Leskovsek, “Wear resistance and anti-sticking properties of duplex treated forming tool steel”, Wear vol. 254, pp. 1113–1121, 2003.

[14] N.Y,Tyurin, O.V, Kolısnıchenko, N.G, Tsygankov,” The Pulse Plasma Technology”, The Paton Welding Journal, vol. 1, pp. 38-43, 2004.

[15] J. Langnera, J. Piekoszewskia, Z. Wernera, U, V.I. Tereshin, V.V. Chebotarev, I. Garkusha, L. Wali´s, B. Sartowska, W. Starosta, W. Szymczyk, M. Kopcewicz, A. Grabias, “Surface modification of constructional steels by irradiation with high intensity pulsed nitrogen plasma beams”, Surface and Coatings Technology, vol. 128-129, pp. 105-111, 2000.

[16] M. Ulutan, O. N. Celik, H. Gasan, U. Er, “Effect of different surface treatment methods on the friction and wear behavior of AISI 4140 steel”, J. Mater. Sci. Technol, vol. 26, pp. 251-257, 2010.

[17] Y.Y. Ozbek, C. Sarioglu, M.Durman,“ The effect of plasma detonation parameters on residual stresses developed in the plasma

modified layer", Vacuum, vol. 106, pp. 11-15, 2014.

[18] Q.F. Guana, H. Zoua, G.T. Zoua, A.M. Wub, S.Z. Haob, J.X. Zoub, Y. Qinb, C. Dongb, Q.Y. Zhang, "Surface nanostructure and amorphous state of a low carbon steel induced by high-current pulsed electron beam", Surface & Coatings Technology, vol. 196, pp. 145– 149, 2005.