INFLUENCE OF HEAT TREATMENT PARAMETERS, TEMPERATURE AND TIME, ON WEAR AND MICROHARDNESS OF NICRBSI FLAME SPRAYED COATINGS APPLIED ON CK45 SUBSTRATES

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Abstract:*The objectives of this research were to find an economical way of reducing porosities in the microstructure of coatings deposited by flame spraying technique on CK45 steel and also trying to increase their cohesive strength to the substrate, so that the overall wear properties of this type of coating can be improved. So several specimens from this steel coated with NiCrBSi powder under specific conditions were subjected to various furnace heat treatment at 1000, 1025, 1050, 1075 and 1100 °C, each for periods of 5, 10 and 15 minute before cooling them in air. Tribological properties of treated coatings were evaluated by pin on disc method. The results show the highest wear resistance and microhardness values observed in one of the sample was due to lower amount of porosity and higher amount of very fine $Cr_2Ni_3B_6$ particles precipitated homogeneously throughout its microstructure during specific heat treatment.*

Keywords: *NiCrBSi coatings, Flame spraying, Coating, Heat treatment, CK45, Wear, Microstructure, Microhardness, Pin on Disc*

1. INTRODUCTION

Coatings applied by flame spraying technique has found a great deal of applications in various industries when highly aggressive environments are involved ,since a wide range of materials have the potential to be used in thermal spraying processes [1-4]. In particular case where high resistance to corrosion and wear are required, self diffusion nickel base alloys are usually used as the main coating ingredients [5-8]. Among these alloys, NiCrBSi alloy has one of the highest applications as an overlay coating material [9-11].

Bonding strength of typical flame sprayed coating contains 10-20% porosities within its microstructure is usually weak. Applications of various heat treatment cycles on the coated samples can reduce the amounts of porosities to 0.3-5% and strengthen the bonding between the splats and between the coating and the substrate. The holding time and the amount of temperature utilized for heat treatment of sprayed coated samples are very crucial for the quality of the final coating microstructure and its tribological properties [1, 5, and 11].

There are published literatures on several mechanical properties [4-6 and 11-12] of NiCrBSi coatings produced by various techniques, but subjected to similar heat treatment cycles. These articles compares the mechanical behaviour of this type of coating that produced by different techniques, however the present research compares some of the mechanical properties of the coatings produced by single technique subjected to various heat treatment cycles. Wear and hardness behaviors of NiCrBSi coatings applied by flame spraying technique and subjected to various heat treatments were investigated and the heat treatment conditions led to optimum wear resistance and maximum microhardness value has been established.

2. EXPERIMENTAL PROCEDURE

NiCrBSi powder with merchandise code PE 3309 was utilized for coating samples of CK45 carbon steel. Chemical positions of both powder used for coating and the substrate are presented in

Table 1. The powder has a spherical morphology with a mean diameter of 33 μm. The steel substrates were in the form of disc with a thickness of 10 mm and diameter of 25 mm. Table 2 presents the constant conditions used for flame spraying. The spraying procedure was performed in such a way that to get the coating thickness of all the specimens within the range of 250-300 μm. The coated samples were treated at 1000, 1025, 1050, 1075 and 1100 °C, each for periods of 5, 10 and 15 minutes before cooled them in air. The coated samples were coded according to Table 3.

Table 1. Chemical compositions of NiCrBSi powder and substrate steel

CK45 substrate	NiCrBSi powder	Weight (%)
≤ 0.40	Balance	Ni
≤ 0.40	15.70	Cr
Balance	4.08	Fe
0.42 - 0.50	0.81	C
≤ 0.40	4.27	Si
-	3.35	B
0.5 – 0.8	-	Mn
0.035	-	P
0.035	-	S
≤ 0.10	-	Mo

Table 2. Constant Parameters used for coating

Parameter	amount
Acetylene feed rate	0.93 m^3/h
Acetylene pressure	100 KPa
Oxygen feed rate	m^3/h 1.7
Oxygen pressure	170 KPa
Torch X-axes motion speed	85 mm/sec
Torch Y-axes motion speed	25 mm/sec

Table 3. Codes of the samples heat treated at various temperatures and times

Heat treatment cycle °C, min, AC*	Sample Code	Heat treatment cycle °C, min, AC*	Sample Code
1000, 5, AC	00-S-5	1050, 15, AC	50-S-15
1000, 10, AC	00-S-10	1075, 5, AC	75-S-5
1000, 15, AC	00-S-15	1075, 10, AC	75-S-10
. 1025, 5, AC	25-S-5	1075, 15, AC	75-S-15
1025, 10, AC	25-S-10	1100, 5, AC	100-S-5
1025, 15, AC	25-S-15	1100, 10, AC	100-S-10
1050, 5, AC	50-S-5	1100, 15, AC	100-S-15
1050, 10, AC	50-S-10		

* Air Cooled

Tribological evaluations of heat treated coatings were carried out by pin on disc apparatus according to ASTM G99-95 standard. Cylindrical pins with a diameter of 4mm made of martensitic steel (HVN=650) were used. The discs were the coated samples under study. The normal load applied was 80N; the rotation speed of pin on disc and the total sliding distance were 0.133 m/s and 500m respectively. The samples were cleaned with acetone and dried before and after wear test; then weighted and the difference between their weights evaluated as wear resistance parameter. The shapes of worn traces and debris were evaluated by SEM. Finally microhardness measurements were carried out on the coating surfaces and coating/substrate cross section. The load and the loading period in this test were 100gf and 15 sec respectively.

3. RESULTS AND DISCUSSION

3. 1. Wear

The result of wear tests of the coated samples treated at various temperatures and times are presented in Fig.1 and Table 4. The wear rate coefficients (k) of the samples were calculated according to the following relationship [14].

$$k = W/LC$$

Where W is the weight loss of coating after wear in milligram, L is the wear distance in meter, C is the normal force applied on the disc in Newton.

According to Fig. 1 and Table 4, the maximum wear resistance (i.e. minimum wear rate) was observed in the sample treated for 5 minute at 1075 °C, other samples treated for 5 minute at lower temperatures had much lower wear resistance relative to this sample. In addition, Table 4 shows the wear rate coefficients of the samples treated for 5 minute at temperature range 1000 – 1075 °C has a large amount of standard deviations relative to other samples treated for 10 and 15 minute at this temperature range. The wear rate coefficient of the samples treated for 5 minute decreased as the temperature increased from 1000 to 1075 °C and then increased slightly at 1100 °C; so that it changed from 5.5×10^{-4} mg/Nm at 1000 °C to 1.4×10^{-4} mg/Nm at 1075°C, while such deviations in wear rate coefficients of the samples treated for 10 and 15 minute was not observed. Table 4 shows the wear resistance of the sample 75-S-5 relative to that of non treated coating and CK45 substrate increased 8.5 and 6.5 times respectively.

Typical ground surfaces of several worn

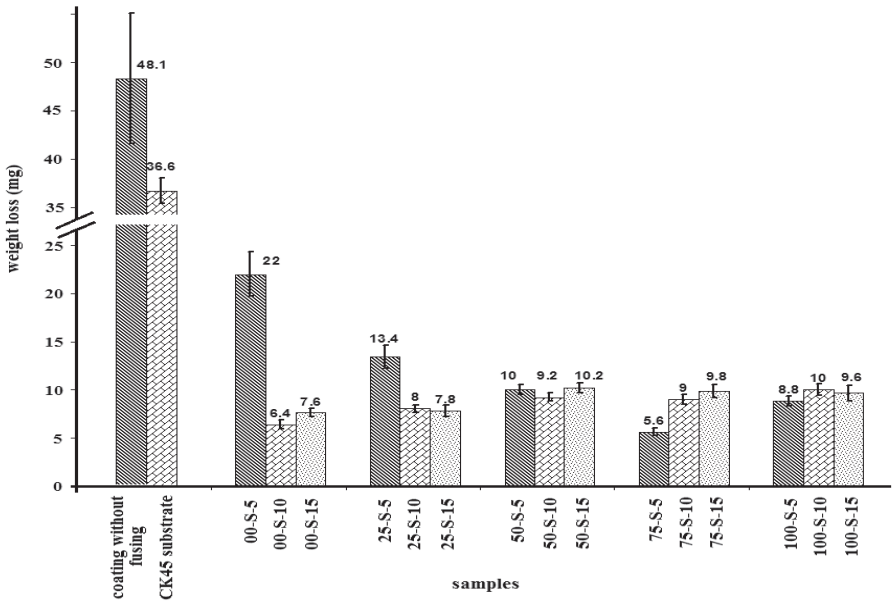

Fig. 1. The weight loss values of the coatings treated at various temperatures and times

Table 4. The average values of wear rate coefficient (WRC) of coatings treated at various temperatures and times

Heat treatment cycle °C, min, AC*	Sample Code	Heat treatment cycle °C, min, AC*	Sample Code
1000, 5, AC	00-S-5	1050, 15, AC	50-S-15
1000, 10, AC	00-S-10	1075, 5, AC	75-S-5
1000, 15, AC	00-S-15	1075, 10, AC	75-S-10
. 1025, 5, AC	25-S-5	1075, 15, AC	75-S-15
1025, 10, AC	25-S-10	1100, 5, AC	100-S-5
1025, 15, AC	25-S-15	1100, 10, AC	100-S-10
1050, 5, AC	50-S-5	1100, 15, AC	100-S-15
1050, 10, AC	50-S-10		

* Air Cooled

samples are presented in Fig. 2 and their corresponding debris of these worn surfaces in Fig. 3. Fig. 3 shows the debris obtained from the sample 00-S-5 are large and have platelet morphology, while those obtained from sample 50-S-5 are smaller and have both polygonal and platelet shapes. The debris obtained from 75-S-5 and 75-S-15 samples were polygonal, however the mean size of 75-S-5 sample's debris was smaller than that obtained from 75-S-15 sample. In addition, some of the worn areas on sample 00-S-5 contain very wide and deep grooves and it seems that they were formed due to pull out of the coating splats from the coated surface, while the groves observed in 75-S-5 and 75-S-15 samples are mainly parallel scratches rather than pull out of splats.

Considering the topography of the worn surface of 00-S-5 sample (Fig. 2a and b), one can observe the splats were pulled out from the

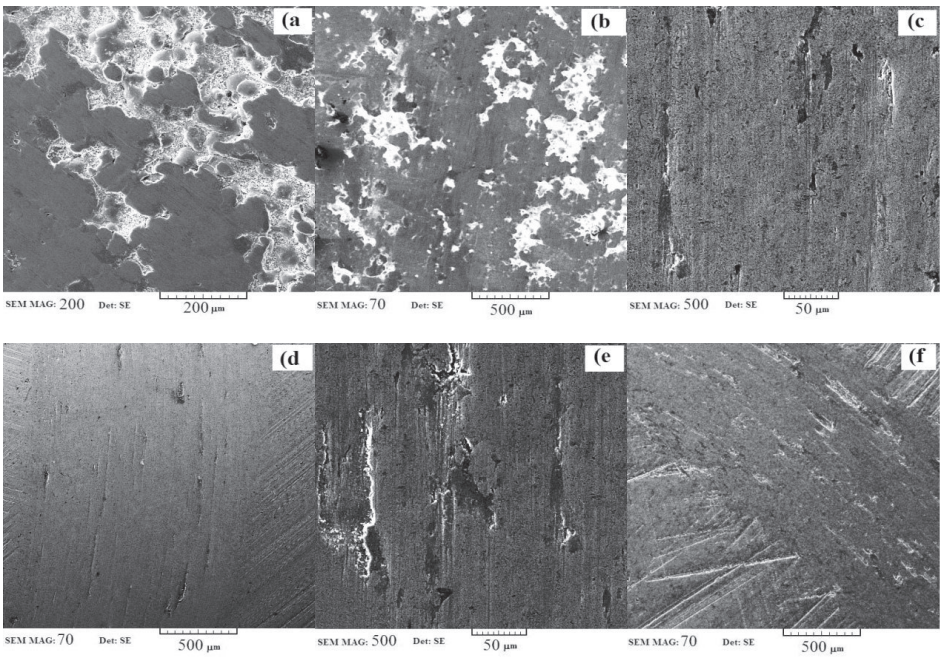

Fig. 2. SEM micrographs of worn surfaces of (a) and (b) 00-S-5, (c) and (d) 75-S-5, (e) and (f) 75-S-15 samples

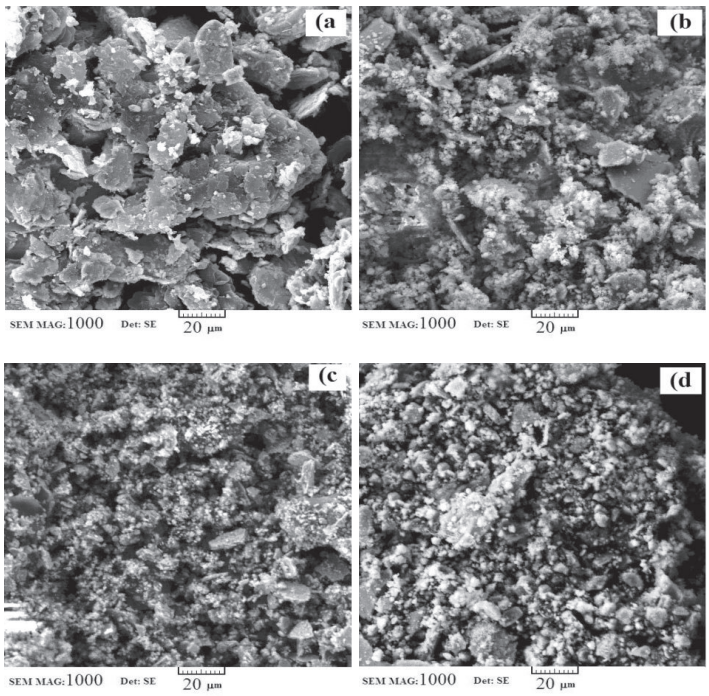

Fig. 3. SEM micrographs of the debris obtained from (a) 00-S-5, (b) 50-S-5, (c) 75-S-5 and (d) 75-S-15 samples

coating surface due to weak cohesive force between the splats. The pull out of the splats during wear test of this type of coating seems to be due to fatigue wear as reported in a similar research by Miguel et al [9]. This mechanism was the dominate wear mechanism in the 00-S-5 sample. On the other hand, the dominate wear mechanism of the sample 75-S-5 seems to be abrasive wear as the parallel scratches observed on the worn surface can be an indication of abrasive wear (Fig. 2c and d). Over the worn coating surface of the sample 75-S-15 parallel scratches can be observed and it seems abrasive wear mechanism was also dominant in this sample.

SEM micrographs of the coatings structures treated at various temperatures and times are showed in Fig. 4. These micrographs show that there is no any boundary between the splats in all of the samples except 00-S-5 and 25-S-5. In addition, for the samples treated for 5 minute at various temperatures, the mean size of the precipitates within coating microstructure increased with increasing temperature. These precipitates were identified as CrB, $(Cr,Fe)_7C_3$, Ni_5Si_2 and Ni_3B .it should be mentioned that CrB and $(Cr, Fe)_7C_3$ were identified by EDS analysis, while Ni_5Si_2 and Ni3B could not be characterized by EDS due to the negligible difference exit between their atomic weight and coating matrix (i.e. Ni), therefore they were identified by XRD analysis. Within the microstructures of the samples treated at 1100 °C for 10 and 15 minute a new phase with a stoichiometric composition $Cr_2Ni_3B_6$ was detected. This phase which was not observed within all other samples formed within the coating in the vicinity of the substrate of 100-S-10 and 100-S-15 samples, Fig. 5.

The mean size of precipitates in 75-S-5 and 75-S-15 samples were 1.2 µm and 3.4 µm respectively. Looking at the debris sizes obtained from the worn surface (Fig. 2) and compare them with the sizes of precipitates observed in the cross section of these coatings (Fig. 4), one may justifies the differences observed in the debris sizes obtained from the worn coatings. The bigger debris produced from 75-S-15 sample is actually due to larger sizes of precipitates existed

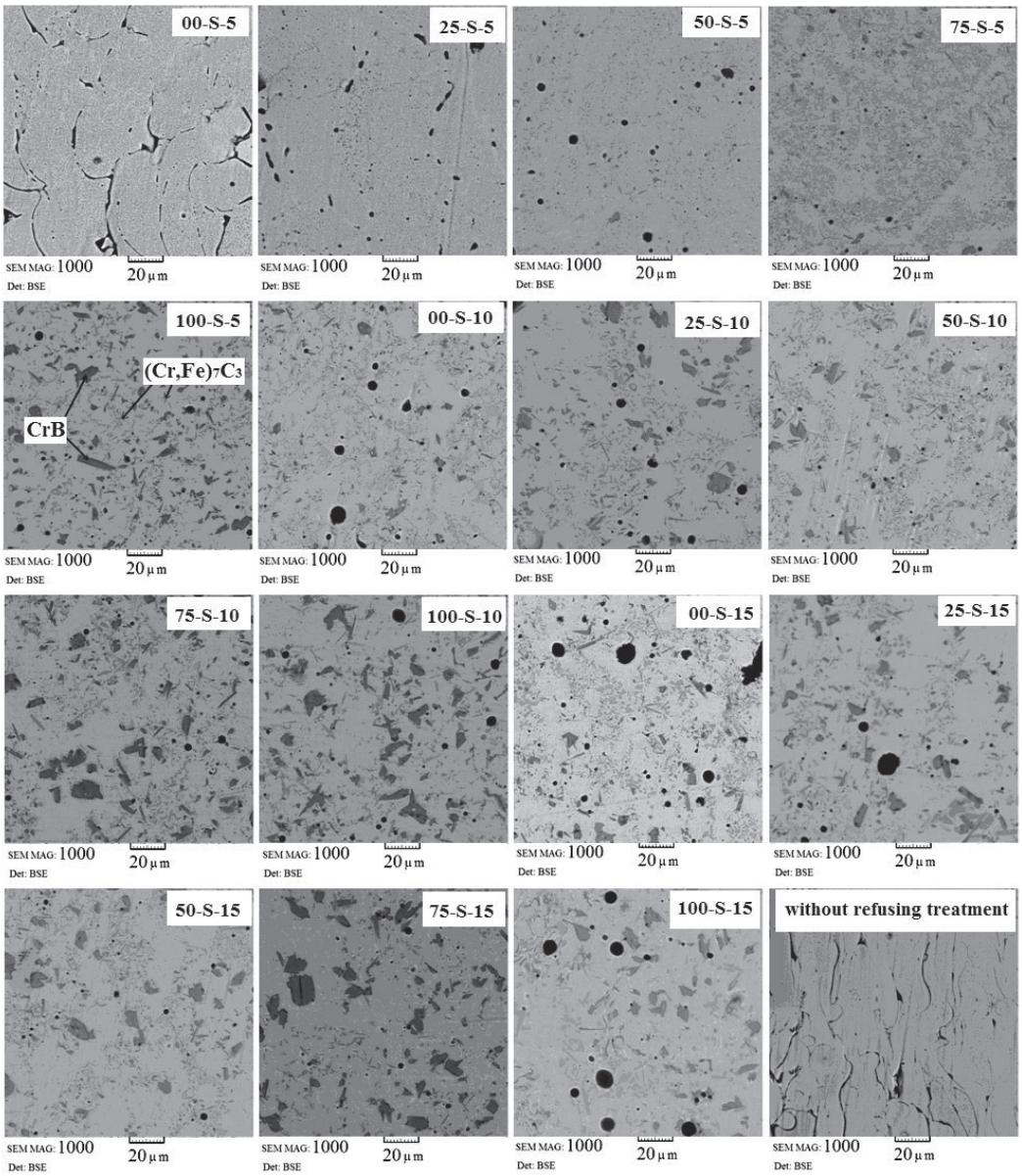

Fig. 4. SEM micrographs of cross sections of the coatings treated at various temperatures and times

within the coating of this sample. This means debris produced during wear test of these samples resulted from pull out of precipitates from the coating.

Fig. 4 also shows the interfaces of splats in the sample without treatment and in the sample 00-S-5 did not vanished completely. This means their splats interfaces are incoherent. As temperature increased from 1000 to 1075°C the amount of coherency increased, so that the improvement in coherency caused an increase in wear resistance of the samples. By increasing the temperature from 1000 to 1075 °C in the samples treated 5 minutes, the mechanism of wear changed from fatigue at low temperature to abrasion at1075°C. On the other hand, when the temperature increased from 1075 to 1100 °C the mean size of the precipitates slightly increased and caused the wear properties to decline an ignorable amount. Therefore one can say that formation of the larger

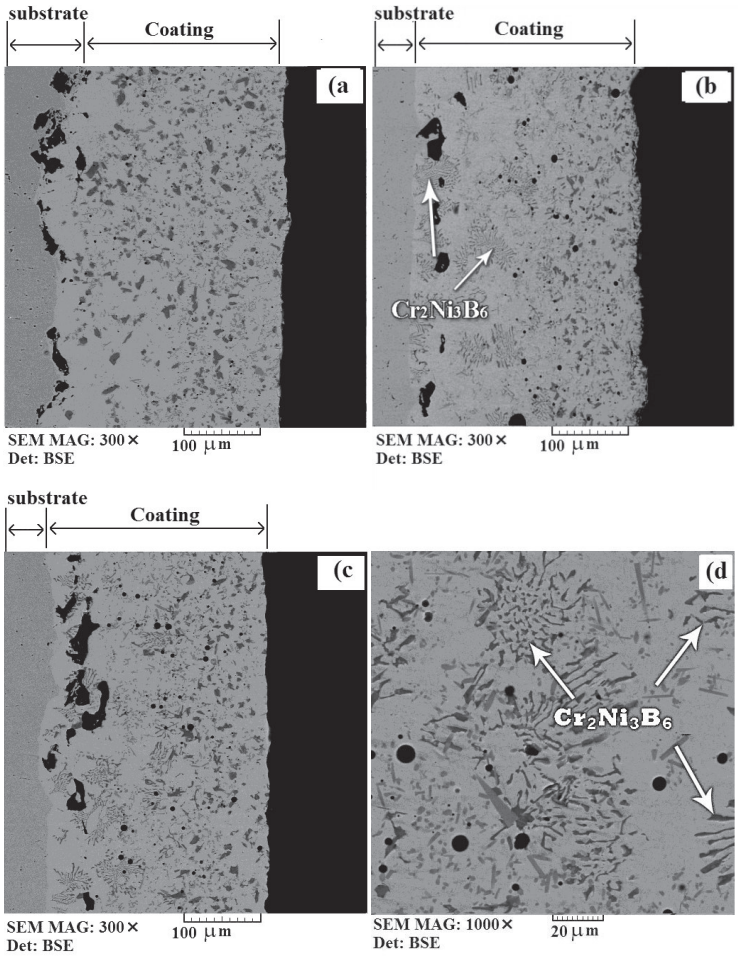

Fig. 5. SEM micrographs of cross section of (a) 75-S-15, (b) 100-S-10, (c) 100-S-15 and (d) 100-S-10 at higher magnification than Fig.4

precipitates larger precipitates within the coating microstructure (Fig. 4) was one of the main causes of reduction in wear resistance. For improving the resistance of wear one should not only increases the coherency between the splats but also reduces the mean size of the coating constituent particles as much as possible. The higher resistance of wear in sample 75-S-5 is basically related to the better coherency between coating splats and smaller precipitates distributed more homogenously within the coating microstructure of this sample.

For a single temperature when the time of heat treatment increased from 5 minutes to 10 and 15 minutes did not cause noticeable effect on wear resistance, however when the temperature increased at a constant treatment time (10 and 15 minutes), the rates of wear slightly increased. Fig. 4 also indicates that the mean sizes of precipitates in all of the samples treated for either 10 or 15 minutes are larger than those treated for 5 minutes and the wear rates of the former samples are higher than those of the later ones. In addition, it can be seen in Fig. 4 that the microstructures of samples 00-S-10 and 00-S-15 are similar to microstructure of sample 75-S-5, their wear rates are approximately similar, and however the wear resistance of 75-S-5 is better than the other two samples. Higher magnification of the coating microstructures of some of the samples in Fig.4 can be seen in Fig.5.

3. 2. Microhardness

Variation of microhardness in coating / substrate cross section for various coated samples

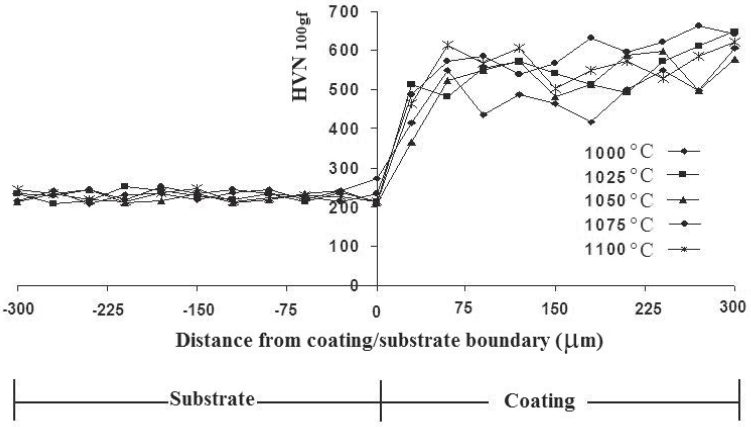

Fig. 6. The mean microhardness profile of substrate/coating cross section of coatings treated at various temperatures for 5 minutes

treated for 5 and 15 minute within the temperature range of 1000 to 1100 °C are presented in Figs. 6 and 7. The microhardness profile in Fig. 6 shows coating of the sample 00-S-5 has the least and that of the sample 75-S-5 has the maximum amount of hardness. This seems to be not only related to the higher amount of precipitated phases and more homogenous distribution of these phases in the sample 75-S-5, but also to the good adhesion of the coating splats in this sample. Since the adhesion of the splats obtained in low temperature treated samples (i.e. 00-S-5) was low, as indicated in the micrograph presented in Fig. 4, the internal energy of the coating was reduced; this resulted to a reduction in resistance against plastic deformation as one can see this reduction in Fig.6.

A very good cohesiveness between the splats of the sample treated at higher temperature (i.e. 75-S-5) was observed; hence higher resistance to plastic deformation was expected for this sample, i.e. higher values of microhardness numbers. Fig. 6 also shows sample 100-S-5 has lower microhardness values than sample 75-S-5, the lower microhardness values in sample 100-S-5 is possible related to the larger precipitates size in this sample in compare with sample 75-S-5.

Fig. 7 shows when the time of heat treatment increased from 5 to 15 minutes only a little change occurred in hardness values, but the sample treated for 15 minute at 1100 °C lost its hardness substantially particularly up to a

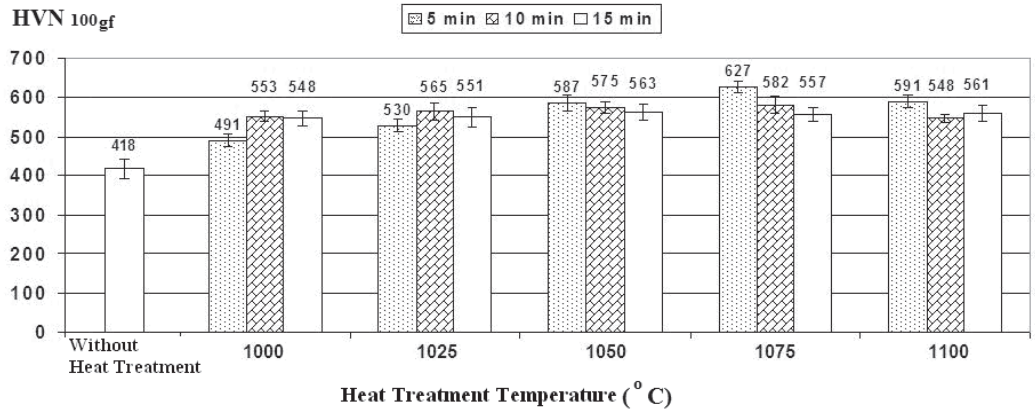

Fig. 7. The mean microhardness profile of substrate/coating cross section of coatings treated at various temperatures and 15 minutes

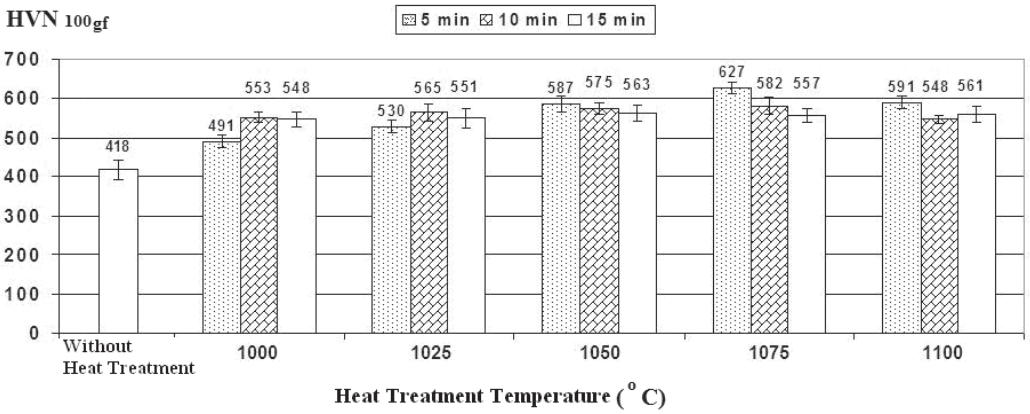

Fig. 8. The mean Microhardness values of coating surface of coatings treated at various temperatures and times

distance of 150 µm from its interface from the substrate. This observation might be related to the formation of $Cr_2Ni_3B_6$ precipitates in 100-S-15 sample that was mentioned in previous sections. The coating microstructure of this sample can be seen in Fig. 5.

A comparison between the mean microhardness values of the coating surfaces is illustrated in Fig. 8. This figure shows the untreated coating surface has the minimum hardness, while the maximum harness obtained at the surface of the coating treated at 1075 °C for 5 minute. The reasons for this behaviour were discussed earlier.

CONCLUSIONS

1. Wear results showed that the sample treated at 1075 °C for 5 minutes has highest wear resistance among other samples.
2. The wear rate coefficients changed from 5.5×10^{-4} mg/Nm at 1000 °C to 1.4×10^{-4} mg/Nm at 1075 °C, while such varieties in wear rate coefficient of the samples treated for 10 and 15 minutes was not observed.
3. Sample treated at 1075 °C for 5 minutes has the highest microhardness due to not only the higher amount of precipitated phases and more homogenous distribution of these phases in this sample, but also due to the good adhesion between its coating splats.

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