Heat treatment of an arc spray NiCr alloy coating after sealing with aluminum phosphate

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ABSTRACT
The paper presents our study on the influence of heat treatment on the properties of an electric-arc thermal spray NiCr20 coating after sealing with aluminum phosphate. The applied annealing temperature was varied in a range of 400-1000^{o}C. Obtained result shows that the porosity of coatings tends to considerably decrease with the increase of the annealing temperature. After treatment at 800-1000^{o}C, over 90% of initial pores in the coating was successfully filled with the sealant. The phase analysis by XRD revealed a formation of different crystalline compounds and an interaction between the coating and the sealant. The filling with the sealant then followed by the heat treatment allowed to increase the wear resistance of the coating by more than 12 times in comparison with the coating without treatment. The ability to increase the resistance of the NiCr20 alloy coatings against corrosion in H_2SO_4 solution by sealing with aluminum phosphate was also investigated. The results indicated the ability of the sealed coating to bring a better protection than the unsealed one in this case.

1 Introduction

The characteristic porous structure of thermal spray coatings not only deteriorates the corrosion resistance of the coatings but also decreases their mechanical properties and consequently the wear resistance [1]. Thus, the coatings require post-spray treatments. One of the most effective treatments is impregnation using polymers, inorganic compounds, or molten metals [1]. Many researches in this field showed that aluminum phosphate sealant can infiltrate deeply into plasma-sprayed coating layers, preventing formation of porosity and increasing their wear resistance [1-5]. The influence of temperature on porosity and wear resistance after sealing with aluminum phosphate has also been mentioned [6-8]. NiCr alloy coatings sealed with aluminum phosphate are widely applied for corrosion resistance at high temperature conditions. Lot of researches indicated that temperature does exert an influence on adhesive ability, physical properties and structural composition of the coatings [9-11]. However, the influence of heat treatment on the characteristics of NiCr20 coating sealed with aluminum phosphate has not been studied yet.

In this study, results concerning the influence of heat treatment on the characteristics of the NiCr20 alloy coatings sealed with aluminum phosphate will be presented. The effect of aluminum phosphate on the corrosion behavior of the NiCr20 alloy coating in H_2SO_4 solution will also investigated.

2 Experiments

- Electric arc thermal spray NiCr20 coating: thickness ~1000 µm.
- Aluminum phosphate solution with 2 major components: reaction between H_3PO_4 85% and $Al(OH)_3$ powder at 120^{o}C, the molar ratio P/Al is 2.3.
- Application of the selant to the NiCr20 coating by brushing, layer-by-layer, until saturation.
- Heat treatment: the coated samples with and without the sealant were under heat treatment for 2 hours at different temperatures varied consecutively from 400 to 1000^{o}C (see **Tab.1**).

Table 1. Labelling of samples

Sample group	Annealing temperature, ^{o}C				
	400	500	600	800	1000
Unsealed NiCr20	NC4	NC5	NC6	NC8	NC10
Sealed NiCr20	NA4	NA5	NA6	NA8	NA10

3 Results and Discussion

Porosity

The unsealed NiCr20 coatings display remaining porosity ranged from 2.5 to 3.9%, which depended on different temperatures of heat treatment (**Tab.2**). The porosity of coating tended to be decreased while increasing temperature. With the NiCr20 alloy coating sealed with aluminum phosphate, the remaining porosity ranged from 1.4 to 2.4%. After treatment with temperature at 800-1000°C, the sealed NiCr20 alloy coatings porosity decreased to 1.4%.

Table 2. Porosity of the sealed and unsealed coatings

Sample	Initial porosity, %	Sealed porosity, %	Remaining porosity, %	Sealed ratio, %
NC4	11.45	7.56	3.89	65.73
NC5	13.13	9.23	3.90	70.42
NC6	14.95	11.31	3.60	75.07
NC8	13.19	9.97	3.21	75.48
NC10	13.83	11.33	2.50	82.11
NA4	13.02	11.09	1.93	84.99
NA5	16.31	13.91	2.40	84.89
NA6	11.93	9.81	2.12	81.63
NA8	15.25	13.84	1.41	90.71
NA10	14.89	13.46	1.43	90.32

Phase composition analysis by XRD

In contrast with aluminum phosphate samples (**Tab.3**), the $AlPO_4$ (Octhorhombic) crystal was not found in the sealed NiCr20 coating samples at 400-600^{o}C → NiCr20 has interaction on the formation of $AlPO_4$ or this phase was still formed but with insignificant amount or at amorphous state (Tab.4).

At 1000^{o}C, the $Ni_3(PO_4)_2$ crystalline phase was formed on the coating surface due to a chemical interaction between the coating and the sealant.

Table 3. Phase composition of aluminum phosphate after annealing at different temperatures.

Annealing temperature, °C	Phase composition					
	$Al_2P_6O_{18}$ Monoclinic	$AlPO_4$ Hexagonal	$H_2AlP_3O_{10}.2H_2O$	$Al(PO_3)_3(N)$	$AlPO_4$ Orthorhombic	$Al(PO_3)_3$ Cubic
400	✓	✓	✓	✓	✓	
500	✓	✓		✓	✓	
600	✓	✓		✓	✓	✓
800		✓		✓	✓	✓
1000					✓	✓

Table 4. Phase composition sealed coating samples after annealing at different temperatures.

Annealing temperature, °C	Phase composition								
	$Al_2P_6O_{18}$ Monoclinic	$AlPO_4$ Hexagonal	$H_2AlP_3O_{10}.2H_2O$	$Al(PO_3)_3(N)$	Ni	$Al_{36}P_{36}O_{144}$ Orthorhombic	$AlPO_4$ Orthorhombic	$Al(PO_3)_3$ Cubic	$Ni_3(PO_4)_2$ Monoclinic
400	✓	✓	✓	✓	✓				
500	✓	✓		✓	✓	✓			
600	✓	✓		✓	✓	✓			
800		✓		✓	✓		✓	✓	
1000					✓	✓	✓		✓

Wear resistance

The wear resistance (as per ASTM G99) of the NiCr20 coating tended to decrease while increasing the annealing temperature (**Fig. 1**). At most testing temperatures, the sealed NiCr20 coating samples exhibited higher wear resistance than unsealed ones. The best wear resistance was observed with the sealed NiCr20 coating after heat treatment at 400°C.

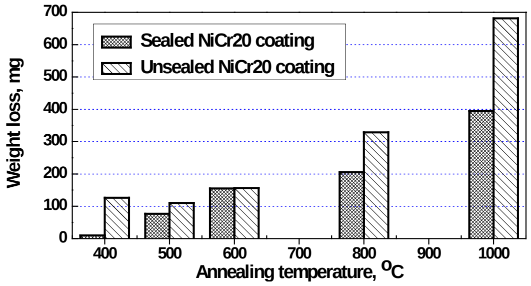

Fig. 1. Wear resistance of coating samples

Corrosion resistance in H_2SO_4 pH 2

The open-circuit potential of sealed coating sample NA4 remained almost stable whereas that of the NC coating reduced from -0.124 to -0417 mV/SCE during 2 hours of immersion in H_2SO_4 pH2 (**Fig.2**). The corrosion current density for NA4 coating is about 3.3 times lower than that for NC coating (**Tab.5**).

Table 5. The corrosion parameters of the coatings after 2 hours of immersion in H_2SO_4 solution at pH 2.

NC			NA4		
R_p (kΩ)	i_{corr} (μA/cm^2)	E_{corr} (mV/SCE)	R_p (kΩ)	i_{corr} (μA/cm^2)	E_{corr} (mV/SCE)
4.69	1.38	-356	28.33	0.42	-272

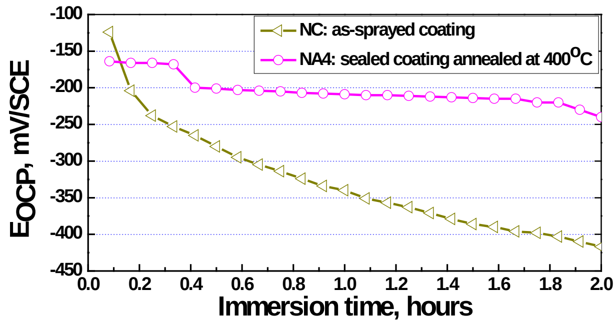

Fig. 2. Evolution of open circuit potential of NC& NA4 samples after 2 hours of immersion in H_2SO_4 pH 2.

4 Conclusions

- The porosity of NiCr20 coating sealed with aluminum phosphate with molar ratio P/Al of 2.3 tends to reduce while increasing the annealing temperature. The ratio of sealed pores reaches more than 90% after the coating was annealed at 800-1000°C.
- The formation of $Al_2P_6O_{18}$ crystalline component in sealed NiCr20 coating is contributed to the improvement of wear resistance. At 1000°C, the sealant has chemical interaction with NiCr20 coating to form the $Ni_3(PO_4)_2$ (Monoclinic) phase.
- The wear resistance of NiCr20 coating has a tendency to considerably decrease with the increase of the annealing temperature. After heat treatment at 400°C, the wear resistance of sealed NiCr20 coating is highest in all of tested samples. It was 12 times higher compared to NiCr20 alloy coating sample.
- The corrosion resistance of NiCr20 alloy coating sealed with aluminum phosphate in H_2SO_4 solution at pH 2 is better than that of the unsealed one.

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