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Use of Aluminium Phosphate for Sealing the Electric Arc Thermal Spray Nickel Chromium Alloy Coating

Nguyen Van Tuan[1*], Le Thu Quy[2], Dinh Thi Mai Thanh[1], Pham Thi Ly[1] and Pham Thi Ha[1]
[1]Institute for Tropical Technology, VAST, 18 Hoang Quoc Viet,
Cau giay, Hanoi, Vietnam
[2]National Key Laboratory for Welding and Surface Treatment Technologies, NARIME, 4
Pham Van Dong, Cau Giay, Hanoi, Vietnam
*Corresponding author: (e-mail: vantuanvktnd@yahoo.com.vn)

The NiCr20 alloy coating on a carbon steel substrate was penetrated by aluminium phosphate sealant at room temperature and under normal pressure for 12 h. Thereafter, the impregnated coatings were heat-treated at 100°C for 2 h, followed by 200°C for 2 h, and at the final curing temperature of 400°C for 4 h. The aluminium phosphate sealant was synthetized with a molar ratio P/Al = 2.3. The analysis on the cross-sectional microstructure by mean of the SEM-EDX revealed that the sealant penetrated into the coating to a depth of 700 µm. After the sealing treatment, about 85% of the pores inside the coating were filled. The XRD phase analysis on the coating surface showed an absence of crystalline compounds between NiCr20 alloy and aluminium phosphate, that meant that there was no reaction between the alloy coating and the sealant. In comparison with the unsealed coating, the application of the sealant improved the coating's wear resistance by 12 times and the microhardness about 25%.

Key words: Aluminium phosphate sealant; thermal spray coating; nickel chromium alloy; wear resistance

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The porosity of NiCr20 coating applied by electric arc thermal spray is very high, normally varied in a range of 10–15% that would impair greatly the protective ability in aggressive chemical environments and the other properties of the coating. There are few methods to reduce the porosity of thermal spray coatings, such as chemical vapour deposition (CVD), chemical vapour infiltration (CVI), metal – organic chemical vapour deposition (MOCVD). The research and application of various inorganic and organic compounds including aluminium phosphate used as sealants for different thermal spray coatings applied by plasma or HVOF methods have been published [1–7]. However, there is still no investigation on the application of aluminium phosphate sealant to electric arc thermal spray NiCr20 coatings.

The physicochemical properties of aluminium phosphate and its pore-filling ability depend greatly on the molar ratio of phosphor and aluminium in its composition. Our recent studies [8] on the influence of the molar ratio P/Al varied in the range of 2–3 on the phase composition of aluminium phosphate by X-ray diffraction (XRD) showed that, in case of molar ratio P/Al = 2.3, there is no residual phase of $Al(OH)_3$ in the phase composition of the aluminium phosphate which affects the viscosity and permeability of aluminium phosphate solution into the NiCr20 alloy coating. This result is consistent with the results given by He *et al.* in 2004 [4]. The properties of NiCr20 alloy coating were penetrated with aluminium phosphate solution with a molar ratio P / Al= 2.3 investigated in this paper: The permeability of aluminium phosphate with a molar ratio P/Al = 2.3 into the NiCr20 alloy coating, and the crystalline phase compounds on the coating's surface after the coating were heat-treated, the coating's porosity and the wear resistance were investigated in this study.

EXPERIMENTAL

The aluminium phosphate was synthetized with 2 major components: orthophosphoric acid (H_3PO_4, 85%) and aluminium hydroxide powder ($Al(OH)_3$)

with a molar ratio P/Al = 2.3 ($Al(OH)_3/H_3PO_4$=1.3/3) at 110°C–120°C.

The NiCr20 alloy coating was prepared by spraying with electric-arc equipment OSU-HESSLER 300A (OSU Hessler GmbH & Co., Germany). The spray parameters for creating coatings were as follows:

- Spray angle: 90°
- Spray distance: 100 mm
- Arc voltage: 33 V
- Current intensity: 200 A
- Nozzles's moving speed: 30 mm/s

The NiCr20 alloy wire with diameter of 2 mm, composition: 79.39%Ni, 18.16%Cr, 0.9%Si, 0.26%Ti, 0.73%Mn, 0.56%Fe (detected by EDX) were used to spray on the C45 steel substrates (diameter of 50 mm, thickness of 3 mm) to obtain the coating. The coating's thickness was about 1000 µm.

The NiCr20 alloy coating on a carbon steel substrate was penetrated by aluminium phosphate sealant at room temperature and under ambiant pressure for 12 h. Thereafter, the impregnated coatings were heat-treated at 100°C for 2 h, followed by 200°C for 2 h, and at the final curing temperature of 400°C for 4 h.

The coating samples after heat-treatment were cut into smaller pieces, epoxy-moulded, grinded, wet-polished with emery papers SiC grades #100 to #2000 and cleaned in ultrasonic cleaning equipment. The samples were then dried in vacuum heating cabinet at 50°C before analysing the cross-sectional microstructure by the optical microscope Axiovert 40 Mat to determine the porosity of the coating [9]. Some analysis by SEM-EDX on the cross-sectional microstructure of the coating were realised to determine the permeability of the aluminium phosphate sealant into the coating.

The phase composition on the coating's surface was examined by X-ray diffraction (XRD) by using D5005 Roentgen analysis equipment with Cu-Kα radiation with the scan step for 2θ at 0.02°/s.

The sliding wear resistance of the coating was determined by using a pin-on-disk TE-91 Precision Rotary Vacuum Tribometer in accordance with A*STM G99:2000 standard*. The test was conducted under following conditions: applied constant load of 30 N, the wear track radius of 20 mm, rotational speed of 382 rpm, sliding speed of 0.4 m/s, duration of 900 s [10]. The microhardness on the surface of coatings was measured by the Vickers hardness (HV) with a loading of 300 g and a retention time of 15 s on the AVK-Co / Mitutoyo hardness measuring equipment.

RESULTS AND DISCUSSIONS

Permeability of the Sealant

Figure 1 shows a SEM cross-section of the NiCr20 coating after sealing with aluminium phosphate sealant. Table 1 shows the EDX results analyzed at 12 different positions of the SEM cross-section of the sealed coating sample. The presence of Al, P and O elements at the positions 010 and 011 in the micrograph showed that the aluminium phosphate solution penetrated to a depth of about 700 µm into the NiCr20 alloy coating.

The Porosity

The porosity of the sealed coating and unsealed coating were determined by optical microscope Axiovert 40 Mat equipment coupled with a multiphase image analysis software. The results of the image analysis at 5 positions on the cross-section of the samples are presented in Table 2.

The results of the image analysis on the cross-section of the samples indicated that the pores of the NiCr20 alloy coating were sealed to a proportion of about 85% by the aluminium phosphate sealant after the heat treatment. The unsealed coating showed a porosity of around 11%, while the porosity of the sealed coating was below 2%. Figure 2 shows a cross-section image of the sealed NiCr20 coating.

Table 1. The results of SEM-EDX analysis at different positions on the cross-section of the coating sample after sealing with aluminium phosphate.

Analysis position	Distance from the surface (µm)	Element content (% w)				
		Al	P	O	Ni	Cr
001	0	3.14	22.01	34.79	21.62	7.01
003	70	2.22	13.20	22.51	46.38	13.59
004	80	0.27		4.48	82.74	12.12
005	147	0.19		0.13	77.99	20.38
006	210	0.61		11.64	60.53	23.54
007	300	0.26		0.51	77.59	20.17
008	434	0.18		0.69	77.81	20.03
009	522	0.15		0.24	78.49	19.67
010	657	5.60	8.04	12.59	61.36	10.24
011	681	7.01	0.06	31.69	3.83	41.99
012	784			1.25	77.60	19.90
002	1200			1.54	79.21	19.25

Table 2. The results of the sealed coating porosity analysis by optical microscopy SEM-EDX analysis at different positions on the cross-section of the coating sample after sealing with aluminium phosphate.

Analysis position	Surface ratio (%)			Proportion of sealed pores (%)	
	NiCr20	Remained pores	Sealant-filled pores	Calculated	Mean
1	87.18	2.42	9.17	79.12	85 ± 3.51
2	*85.13*	*2.13*	*11.45*	*84.32*	
3	86.71	1.48	10.26	87.39	
4	84.05	1.78	12.08	87.16	
5	83.94	1.87	12.49	86.98	

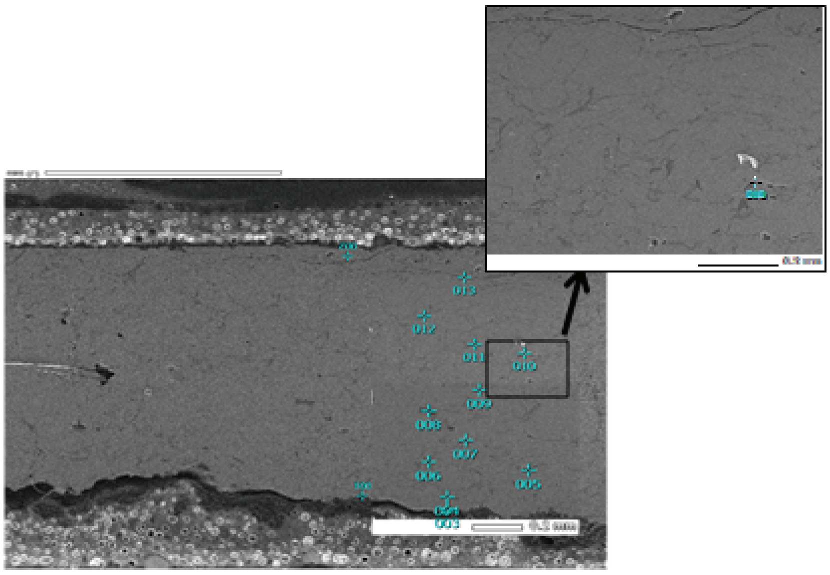

Figure 1. The SEM-EDX analysis positions on cross-section of the NiCr20 alloy coating after sealing with aluminium phosphate with molar ratio P/Al = 2.3.

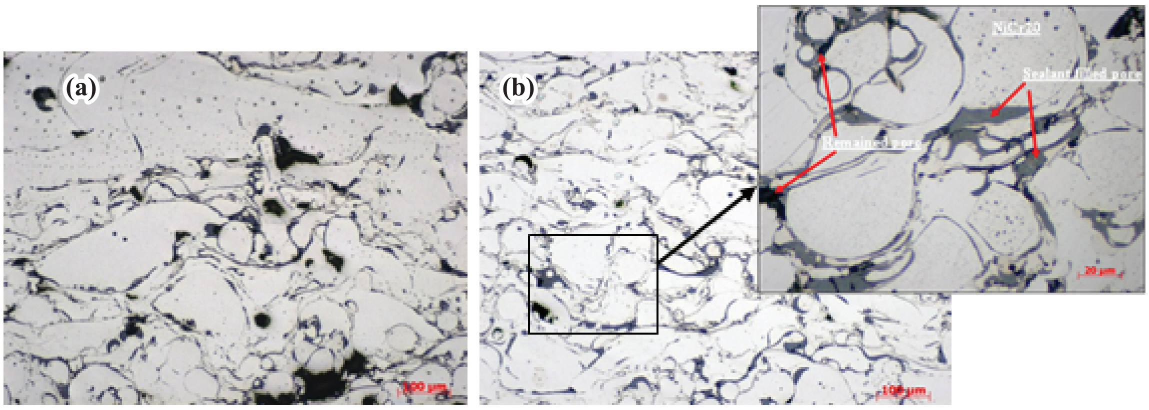

Figure 2. The microtructure of cross-section of the NiCr20 alloy coating before (a) and after (b) combined with aluminium phosphate sealant.

Phase Composition

Figure 3 shows a XRD spectra realised on the surface of the NiCr20 coating after sealing with aluminium phosphate sealant.

The results of the XRD analysis indicated that the coating after heat-treatment was a mixture of several phases: Ni, ($H_2AlP_3O_{10}.2H_2O$), $Al(PO_3)_3$, hexagonal $AlPO_4$, and Monoclinic $Al_2P_6O_{18}$. The results of the XRD analysis of the coating surface showed an absence of crystalline compounds between NiCr20 alloy and aluminium phosphate, that meant that there was no reaction between the alloy coating and the sealant. The formation of metaphosphate phase of $Al(PO_3)_3$ and $Al_2P_6O_{18}$ may increase the abrasion resistance of the thermal coating [11,12,13].

Wear Resistance

The weight loss method was used to determine the wear resistance of the coating samples after treatment. The results of wear resistance tests are shawn in Figure 4.

The higher wear resistance measured on the

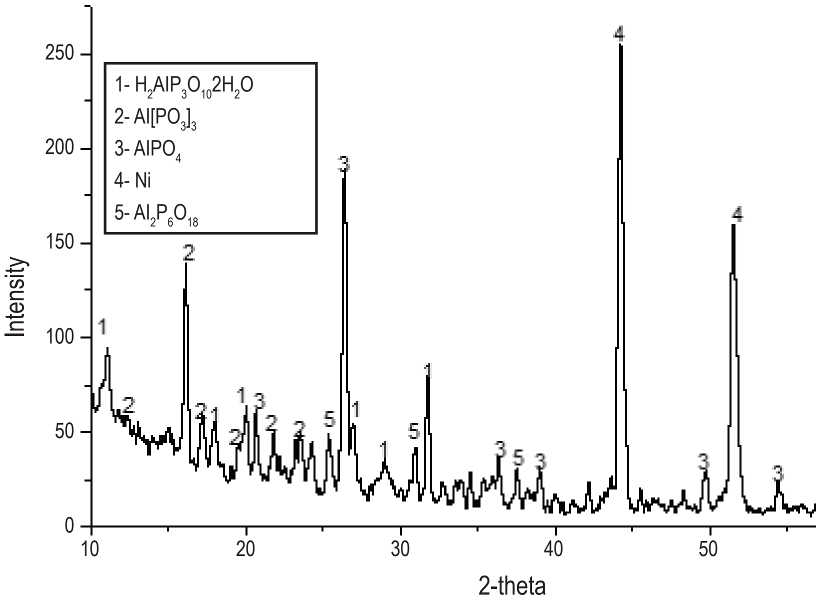

Figure 3. XRD patterns of the NiCr20 alloy coating sample sealed with aluminium phosphate solution after heat treatment at 400°C.

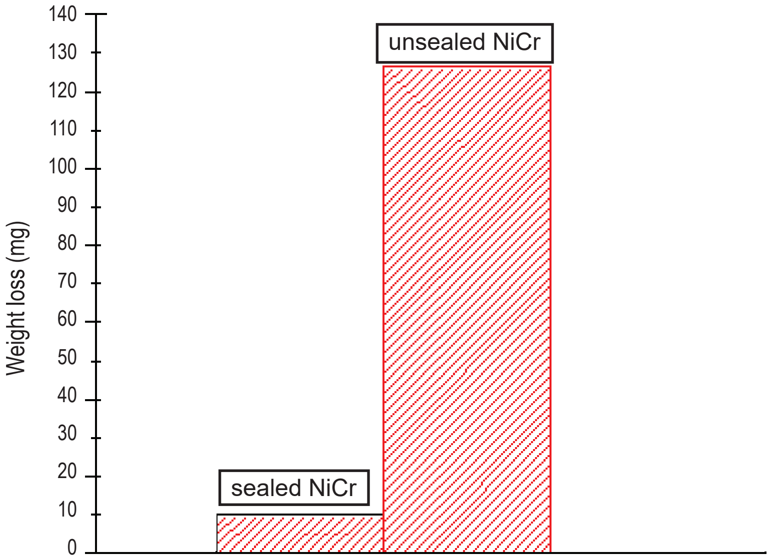

Figure 4. Wear resistance of the sealed and unsealed NiCr20 alloy coating samples.

sealed NiCr20 alloy coating was in good accordance with the result obtained by the phase composition analysis. In comparison with unsealed coating sample, the existence of the crystalline phase $Al_2P_8O_{16}$ in the sealed coating sample contributed to considerably increase the wear resistance. Moreover, the porosity of the unsealed coating was about 11%, i.e. much higher than the porosity of the sealed coating (around 2%). The existence of pores in the coating was one of the reasons to reduce the abrasion resistance. After the wear test, the weight loss of the sealed NiCr coating was 9.8 mg, whereas that was 126.3 mg for unsealed coating. Thus, in comparison with the unsealed coating, the application of the sealant improved the coating's wear resistance by 12 times.

Table 3. Microhardness of the coating.

No.	Sample	Microhardness HV 0.3, 15 s
1	The unsealed coating	267
2	The sealed coating	315

Microhardness

Results of the microhardness measurement are presented in Table 3.

The results indicated that the hardness of the sealed coating increased about 25% in comparison with the unsealed coating. The high hardness of sealed coating is due to the formation of metaphosphate phase of $Al(PO_3)_3$ and $Al_2P_6O_{18}$ in the coating [7].

CONCLUSION

Aluminium phosphate sealant had significantly improved some properties of NiCr20 coating such as reduced porosity, increased the wear resistance and microhardness of the coating. The results of this study showed the effectiveness of using aluminium phosphate sealant for arc thermal spray.

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