



Effect of Surface Morphologies on the Isothermal Oxidation Behavior of MCrAlY Coatings Fabricated by High-velocity Oxyfuel Processes

M. Tahari^a, F. Naeimi^{*b}

^a Department of Engineering, Esfarayen University of technology, Esfarayen, North-Khorasan, Iran

^b Department of Engineering, Isfahan (Khorasgan) Branch, Islamic Azad University, Isfahan, Iran

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ABSTRACT

In this study, the effect of surface morphologies on the isothermal oxidation behavior of MCrAlY coatings fabricated by high-velocity oxyfuel (HVOF) processes was investigated. To do so, free standing coatings with as-sprayed and polished surfaces were isothermally oxidized at 1050°C. The thickness of the bond coat was approximately 380µm. The Ra of the as-sprayed and polished surfaces were 6.4µm and 1.1µm, respectively. Before and after oxidation, the surface scale composition and morphology were analyzed by scanning electron microscope (SEM/EDX) and X-ray diffraction. The results show that the roughness of the bond coat significantly affected oxide scales formed on the as-sprayed and polished coatings after 100h oxidation. With increasing roughness (Ra) of the bond coat, the oxidation rate constantly increased and composition of thermal growth oxidation (TGO) changed into oxide mixture consisting of Cr₂O₃, NiO and spinel. In addition, Scanning Electron Microscope analysis showed that thickness of TGO Layer of the polished CONiCrAlY coating at 1050°C was much thinner than that of the as-sprayed one.

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1. INTRODUCTION

Thermal barrier coatings (TBCs) are widely used for oxidation and corrosion protection of hot section components, such as blades, combustors and vanes in modern stationary gas turbines engines. TBCs increased inlet temperatures and thus improve engine efficiency and performance [1]. TBCs generally consist of a ceramic top coat, a TGO, and metallic bond coat on the nickel-base superalloy substrate. MCrAlY (where M stands for Ni, Fe, Co or some combination of them) is commonly used as a metallic bond coat. The bond coat layer is normally applied by low-pressure plasma spray (LPPS), high frequency pulse detonation (HFPD), vacuum plasma spray (VPS), air plasma spray (APS), and HVOF [2]. HVOF spraying has been more recently used because compared to LPPS or VPS, it can be used at atmospheric pressure, and thus, it costs less, while its quality is higher than VPS or LPPS systems operated in

vacuum [3]. During services at elevated temperature, a TGO layer is formed at the surface of the band coat. The growth rate of the TGO layer assists the increase of tensile stresses and the growth of horizontal cracks at the surface of the band coat. The elements in the bond coat can also form oxides such as: Cr₂O₃, NiO, CoO and spinel oxides (NiAl₂O₄, CoAl₂O₄) which are not as protective as alumina. Composition of TGO layer plays an important role in the lifetime and failure of TBCs [4, 5]. Alumina, which is formed on the bond coat can offer good protection due to its slow growth rate, excellent adhesion to ceramic top coat and high chemical barrier to oxygen transport and thermodynamic stability. It is desired that this oxide scale be a continuous, thin, uniform and dense α-alumina layer [6]. The microstructure of the thermally grown oxide depends on the services condition, coating composition and deposition method [7-10]. Many Studies were carried out to improve the bond coat oxidation resistance by modifying the surface of bond coat. Investigations have shown that surface condition of bond coat has a serious effect on the microstructure of TGO [11, 12]. In [13],

*Corresponding Author's Email: farid.naeimi@gmail.com (F. Naeimi)

the authors found spinel oxides in the case of as-sprayed samples; however, in the polished and ground samples, oxides like NiO, CoO and α -Al₂O₃ were found. In [14], the authors found that the oxides formed on a rough surface showed a different morphology as compared to those on a flat surface. In [15], the authors found that the scale formed on the grit-blasted coating was a large decreased in growth rate compared with the as-sprayed coating. Therefore, studying the effect of surface condition on the isothermal oxidation behavior was carried out and the results have been reported.

2. EXPERIMENTAL PROCEDURE

In this study, Inconel 738 Superalloy was used as a substrate. The CoNiCrAlY bond coat was deposited on sand blasted Inconel substrate using HVOF spray technique. The HVOF spraying was carried out using a commercial Met Jet III (Metallization) gun. Kerosene and Oxygen were used as fuel and carrier gas, respectively. HVOF spray conditions were reported in Table 1. The chemical composition of the used CoNiCrAlY powder is presented in Table 2. The thickness of MCrAlY bond coat was about 300-400 μ m. Depending on sample surface treatment, two classifications were used (A and B).

TABLE 1. HVOF deposition conditions.

Spray distance (cm)	40
Powder feed rate (g/min)	50
Fuel flow rate (ml/min)	260
Oxygen flow rate (ml/min)	800

TABLE 2. Chemical composition (wt.%) of the CoNiCrAlY powder.

Co	Ni	Cr	Al	y
bal.	33	19	9	0.5

The surfaces of samples that were as-sprayed are denoted as type A, while the other samples' surfaces were polished with 600 μ m- SiC paper and denoted as type B. Quantitative measurements of the surface roughness were made using a surface profile recorder (PS1, Mahr Group). The surface roughness was measured on each coating in more than three locations, and the average value was used to determine the surface roughness. The isothermal oxidation tests were carried out at 1050°C in a heated furnace in air. Samples were placed in an alumina crucible and kept in the constant temperature zone of the furnace at 1050°C for different periods of time. The specimens for oxidation tests were

then taken out for measurement of the mass change at intervals by an electronic balance with an accuracy of ± 0.0001 g. Three measurements for mass gain at each time were taken and averaged. Before and after oxidation, the surface of the samples was examined by scanning electron microscopy and energy dispersive X-ray analysis (SEM/EDX-PHILIPS XL-30) to determine the scale morphology and chemistry as well as by X-ray diffraction (XRD) technique for phase identification.

3. RESULTS AND DISCUSSION

One of the important parameters which can affect the quality of the coatings resulted from the HVOF process are morphology, distribution, and size of the powder particles. Powders with a spherical morphology usually offer the best quality. The more uniformly the powder particles are distributed, the more likely the quality of the coating is improved. Scanning electron microscope (SEM) images of the powder used in this study are shown in Figure 1.

It was observed that the dimensions of the powder used in the study were 20-25 μ m and the spherical morphology was truly appropriate for HVOF thermal spraying. The given powder with the parameters mentioned in the part methodology was sprayed onto the substrate. The surface morphology after spraying (A), and polished (B) are shown in Figure 2.

During the thermal spraying, the powder particles appear on the coating melted, semi-melted, and unmelted, depending on the conditions and parameters of spraying including the size and morphology of the powder, substrate hardness, temperature, velocity, and kinetic energy of the powder particles [16, 17]. As it is observed, the powder particles approximately kept their original shape and the coating was covered by quite spherical particles. However, in the polished sample, removing the spherical particles from the surface led to the creation of smooth surfaces on the coating.

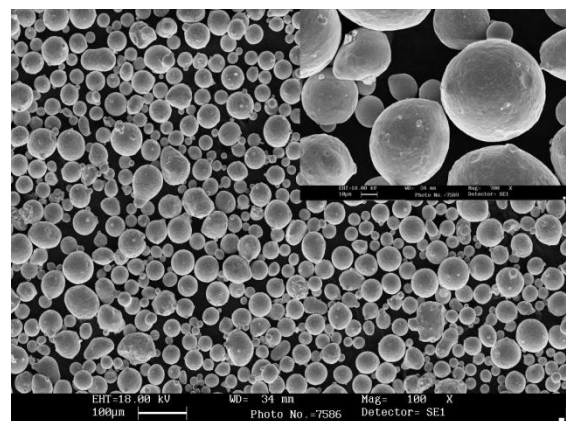


Figure 1. SEM micrographs of the CoNiCrAlY powder

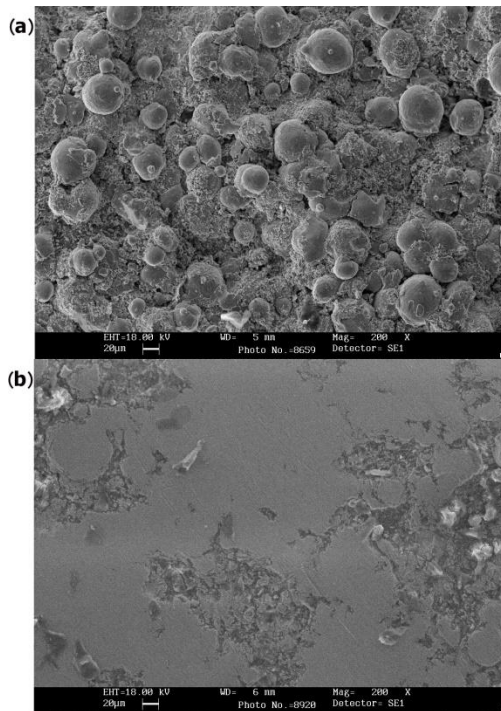


Figure 2. Surface morphology of the HVOF-sprayed coating a) sample A (as-sprayed) b) sample B (polished).

This has led to the fact that the special coating surface was reduced under the influence of the polishing process.

Figure 3 illustrates the x-ray pattern of the coating. To better investigate the effect of the HVOF process on the structure of the coating, the x-ray pattern of the powder used in the study is shown as well. Comparison between the X-ray pattern of powder and coating revealed that the peak widths of phase γ in the coating X-ray patterns were more than that of the powder. Some researchers like Richer and Zhao [18, 19] carried out research on thermal spraying of the commercial powder CoNiCrAlY using Cold Gas Dynamic Spray (CGDS) and reached similar results. They maintain that fine grains, and internal stresses have led to the widening of the peaks.

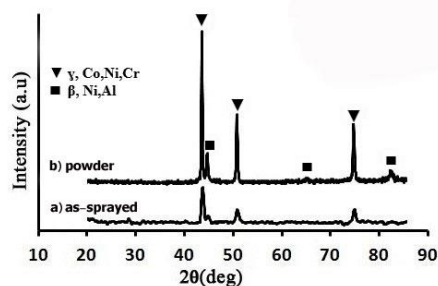


Figure 3. X-ray diffraction patterns of a) MCrAlY coating b) MCrAlY powder

Considering the findings, it is clear that temperature, velocity and the kinetic energy of powder grains, as well as melting or unmelting of the powder grains during spraying can be effective factors of the grain size of the coatings.

Figure 4 shows the cross section of the coating. As it can be seen, the coating is about 380 μ m wide which is an optimal width for thermal spray coatings.

In a study on thermal spraying of the alloy Ni-Cr using HVOF, Saeedi et al [20] reported that a black stratum was formed around the splats during thermal spraying of the metals sensitive to oxidation. These black strata were the oxides of the metal formed due to alloying elements oxidation created during the spraying. Toma et al [21] also showed that the oxides formed while spraying MCrAlY were aluminum oxide and Al_xY_yO_z spinel oxide. However, the black oxide strata were not observable at all between the splats in BSE images of the present coating. This implied that appropriate parameters had been applied for the coating of the powder in a way that the powder particles did not express any sign of oxidation during spraying. Pull-off adherence test was done on the samples to investigate coating adherence to the sub-stratum. The findings revealed that adhesion strength of the coating was about 47 MPa, which is consistent with the findings of Li et al. [22]. They showed that coating adhesive strength of MCrAlY created by HVOF thermal spraying was 40-50 MPa. This strength stems from the mechanical locks between splats and the surface roughness of the substratum and can increase as the surface roughness of the substratum increases. The investigation of the interface zone showed that coating and substratum enjoyed a good adherence to the substratum and the mechanical locks between the coating and the substratum roughness had properly linked with one another. Using image analysis software, the porosimetry test was conducted, which showed that the coating porosity was less than 1%. This is an ideal porosity for the thermal spraying coatings using HVOF. The investigations revealed that the morphology of the thermal spraying powder has a strong effect on the coating porosity.

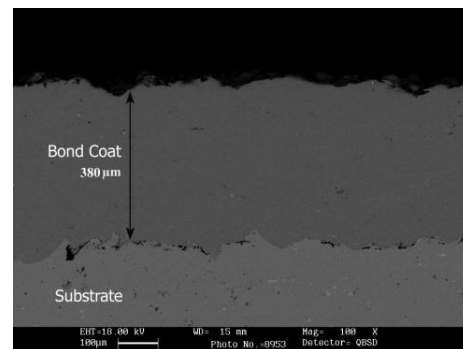


Figure 4. SEM cross-section of the CoNiCrAlY coating

Powders with spherical morphology induce the least porosity and the best coating [22].

In order to investigate the coating surface roughness prior to oxidation, surface roughness profiles were taken from the coating surface. The images of these surface roughness profiles are shown in Figure 5.

As it is observed, both samples have nearly uniform roughness morphology. However, the polished sample is less rough. Different variables are used to illustrate the roughness of surfaces. Due to the fact that the roughness of the surfaces in the samples under investigation was almost uniform, it was decided that Ra criterion was used. This criterion indicates the mean roughness in the surfaces under investigation. The values obtained for the coating roughness after surface treatment are reported in Table 3.

After surface treatment of the coatings, oxidation test was run on the coatings based on the process explained in the part methodology. The results revealed that samples were faced with weight gain during oxidation. The mean of weight gain was finally reported as the coat weight gain. Figure 6 indicates the chart of weight gain based on the time of oxidation.



Figure 5. Surface profiles of MCrAlY coating a) Sample A (as-sprayed) b) Sample B (polished).

TABLE 3. Surface roughness of the bond coat

Sample	Ra (μm)	Oxidation Rate (k_p) 50 Hours	Oxidation Rate (k_p) 100 Hours
A (as-sprayed)	6.4	4.19×10^{-13}	1.49×10^{-13}
B (polished)	1.1	0.21×10^{-13}	0.09×10^{-13}

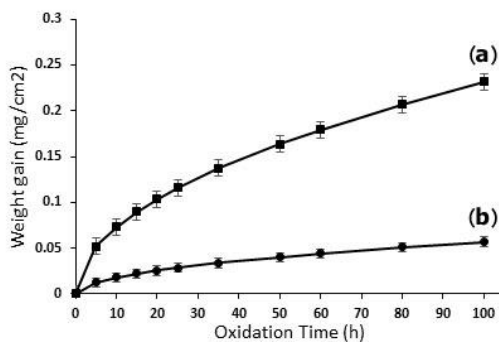


Figure 6. Weight gain measurements as a function of oxidation time at 1050 °C a) Sample A (as-sprayed) b) Sample B (polished)

It is observed that the oxidation rate in both samples was initially rapid and decreased gradually with the passage of time. This type of contribution is related to the cases in which a uniform and dense oxide layer is formed on their surfaces due to the oxidation which subsequently prevents the more penetration of oxygen. With the passage of time, since the thickness of this oxide layer is increased, oxygen penetration worsens. Thus, oxygen rate decreases [23-25]. This kind of curves is called parabolic. Mahesh et al. [26] believed that this phenomenon occurs as a result of oxidation of the coating surface which does not have a protective oxide layer. Moreover, grain boundaries can be places where oxygen may penetrate the coating substrata. However, with the passage of time, due to the growth of oxide layer, the mentioned boundaries are nearly impeded, which leads to the fact that the oxidation rate decreases. To better investigate for different coatings, k_p values or oxidation rates were obtained. The k_p values are reported in Table 4. It is observed that the rate of oxidation in the less rough coating was much less than others. To investigate the reason of this matter, phase and elemental analyses were carried out on the given oxide layer which is discussed later.

To investigate the oxide phases which are created on the surface during the oxidation test and made the TGO layer, XRD test was run on the coating surface. X-ray pattern for both samples under investigation is shown in Figure 7. The comparison of the x-ray patterns of the two coatings indicated that the oxide layer formed in the less rough coating was mostly $\alpha\text{-Al}_2\text{O}_3$.

TABLE 4. The values of oxidation rate (k_p) for coating oxidation in 1050 °C

Coating	Lt	Lc	Rz (μm)	Ra (μm)
Sample A (as-sprayed)	5.600	0.800	32.5	6.4 ± 0.5
Sample B (polished)			11.7	1.1 ± 0.1

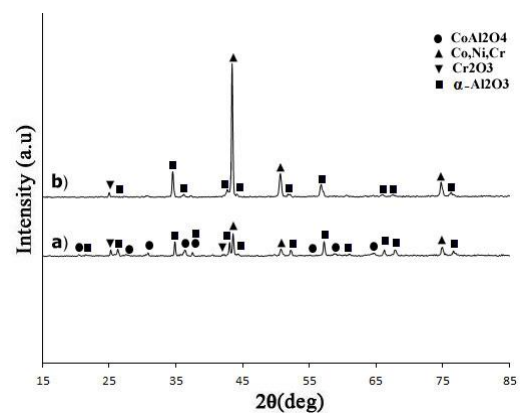


Figure 7. The XRD patterns from (a) the as-sprayed and (b) the polished HVOF CoNiCrAlY coating surfaces after oxidation at 1050°C for 100 h. a) Sample A (as-sprayed) b) Sample B (polished).

With the passage of time, the intensity of the peaks associated with aluminum oxide increases and other phases including spinel are not created in this coating. However, in the more rough coating, it is evidently clear from the x-ray pattern that at the beginning of the oxidation, the oxide layer formed on the coating surface is nearly composed of Al_2O_3 . However, with increasing oxidation time, other phases including chromium oxide and spinel oxides CoAl_2O_4 are also found in it. During oxidation process, other elements in the bond coat can lead to the creation of other oxides including nickel oxide, chromium oxide and spinel oxides, none of which has the ideal condition of alumina layer [27, 28].

To have a closer look at the phases formed during oxidation of the coating surface, EDS test was run on the oxide layers. Figures 8 and 9 illustrate the results of EDX test in 100 hours for the coating with the surface smoothness of $1.1\mu\text{m}$ and $6.4\mu\text{m}$, respectively. As it is observed, in coating with low roughness, the main element of the oxide layer is Al and the oxide layer is fully uniform. However, in the coating with high roughness, two types of oxides are indicated. Running EDS test on this oxide indicated that the substrate included oxides full of Al and the upper layer was made of layers full of Al, Cr, and Ni. Comparison of these results with those obtained from XRD test indicated that the upper oxide layer was composed of spinel oxides which were not accounted as appropriate oxide phases in MCrAlY coatings and enjoyed low surface protective ability [17].

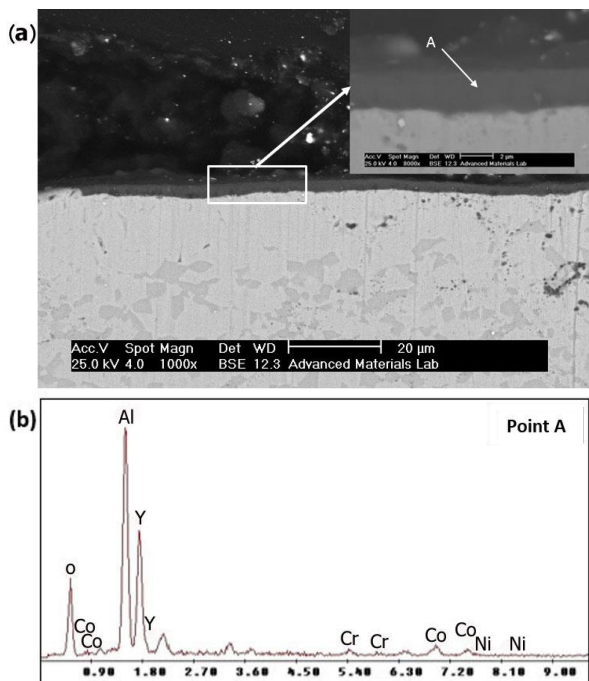


Figure 8. a) SEM cross-section morphology of TGO (as-sprayed) b) the point scanned EDS result of TGO after oxidation at 1050°C for 100 h

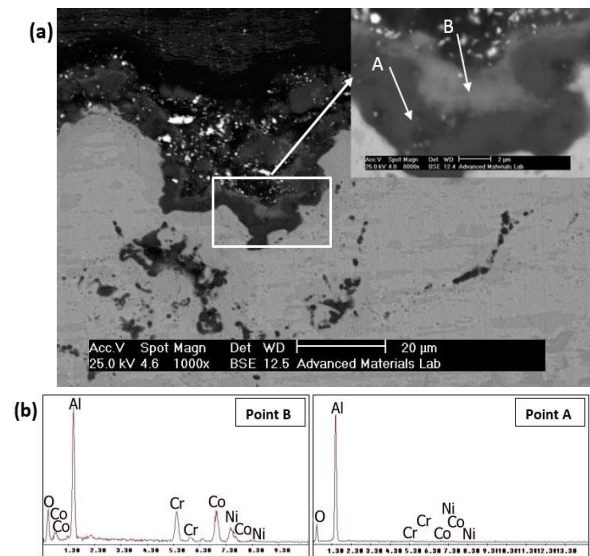


Figure 9. a) SEM cross-section morphology of TGO (as-sprayed) b) the points scanned EDS results of TGO after oxidation at 1050°C for 100 h.

Scholars believe that one of the important factors which determine the type of the formed oxide phases is oxygen activity on the coating surface. In a study done on the MCrAlY nanostructure coating, Mercier et al. investigated the thermal stability of the oxides formed in MCrAlY coatings at different temperatures and concentrations of oxygen using Factsage software.

It is observed that at 1000°C , in oxygen with low activity, Al oxide phase is stable. However, with the increase of the activity of oxygen in the environment, spinel phase shows more stability [29]. Cho et al. believe that when oxygen activity increases in areas with no Al and Cr, CoO and NiO are formed. These oxides are unstable at 1000°C . Thus, they react with Cr_2O_3 and Al_2O_3 and make spinel phases with the overall composition $\text{Co,Ni(Cr,Al)}_2\text{O}_4$. What has been stated about the phase stability was related to the thermodynamic discussion on the oxide formation. However, to investigate oxidation phenomenon, kinetic of the reactions must also be taken into consideration. Due to the fact that Al oxide rapidly reacts with oxygen at 1000°C because of its high activity, it is the first formed oxide on the MCrAlY coatings. It was indicated that this oxide layer showed proper thermal stability in the oxygen with low activity. Hence, if the Al oxide formed on the surface is adequately dense and can impede oxygen penetration to the lower layers and oxygen activity is low in the lower layers of the oxide layer, spinel phases cannot be formed [30]. In the MCrAlY coatings, in the spraying state, the surface topography is one of the effective factors, which can create different thermal oxide layers. Figure 10 indicates the schematic of the effect of the surface morphologies on the preferred areas of spinels.

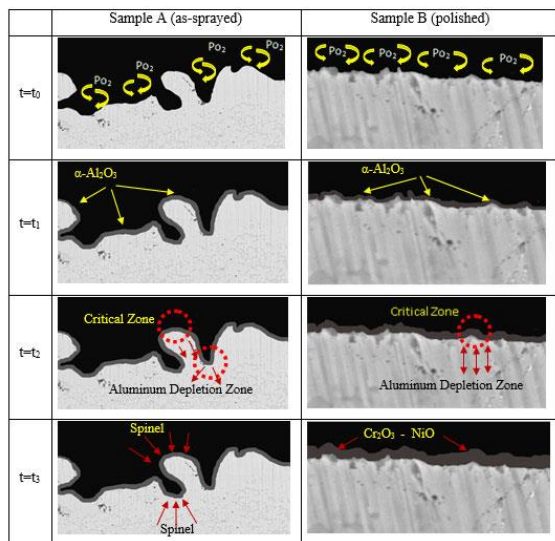


Figure 10. The schematic of the effect of the Surface morphologies on the preferred areas of spinel and other oxides a) Sample A (as-spray) b) Sample B (polished)

This type of surface morphologies in some areas can increase the oxygen activity and decrease Al activity. With the penetration of oxygen in the pores on the surface, alumina layer is created. Then, with the reduction of Al, as the oxygen activity is still high, alumina is converted to other oxides including spinel. Accordingly, in the areas where the oxygen atoms are trapped, numerous spinel phases are formed. In a similar study, Ni et al. indicated that high surface roughness could increase the speed of formation of the unwanted spinel phases [15].

4. CONCLUDING REMARKS

In this study, oxidation of CoNiCrAlY coating under the influence of surface conditions of the bond coat at 1050°C in different times was investigated. Prior to running the oxidation test at high temperature, to obtain different surface conditions, polishing process was used. Using polishing process leads to a reduction in the surface roughness (the Ra of the as-sprayed and polished surfaces were 6.4µm and 1.1µm, respectively) and removal of dents and bumps resulted from the thermal spraying process. The results of the oxidation test indicated that uniformity in the surface conditions led to the uniform distribution of the oxygen activity and formation of dense and continuous layer α -Al₂O₃ on the surface. Moreover, the results revealed that with the increase of surface roughness due to the other oxides including NiO, Cr₂O₃ and spinel, inhomogeneous and non-uniform thermal oxide layer was created and oxidation was constantly increased.

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M. Tahari^a, F. Naeimi^b

^a Department of Engineering, Esfarayen University of technology, Esfarayen, North-Khorasan, Iran

^b Department of Engineering, Isfahan (Khorasan) Branch, Islamic Azad University, Isfahan, Iran

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Surface roughness

High-velocity Oxyfuel

در این پژوهش هدف بررسی تاثیر شرایط مورفولوژی سطحی بر رفتار اکسیداسیون ایزوترم پوشش‌های فلزی MCrAlY اعمال شده به روش HVOF بود. جهت انجام این امر پس از اعمال پوشش، مورفولوژی سطح نمونه‌ها توسط فرایند پولیش تغییر داده شد و تاثیر آن بر رفتار اکسیداسیون آنها در دمای ۱۰۵۰ درجه سانتی‌گراد مورد ارزیابی قرار گرفت. ضخامت پوشش اعمال شده در حدود ۳۸۰ μm و زبری سطح لایه میانی برای حالت پائین شده و پولیش شده به ترتیب ۶٫۴ و ۱٫۱ میکرومتر بود. برای تعیین مشخصه‌های ساختاری و فازی پوشش و لایه اکسید حرارتی (TGO) از میکروسکوپ الکترونی روبشی (SEM/EDX) و آنالیز پراش پرتو ایکس (XRD) استفاده شد. نتایج نشان داد زبری سطح یکی از پارامترهای موثر بر ترکیب شیمیایی لایه اکسید حرارتی ایجاد شده پس از قرارگیری نمونه به مدت ۱۰۰ ساعت می باشد. با افزایش زبری سطح شاهد تغییر ترکیب شیمیایی لایه اکسید حرارتی از ترکیباتی همچون NiO و Cr₂O₃ به اسپینل خواهیم بود. به علاوه نتایج آنالیز میکروسکوپ الکترونی از سطح مقطع پوشش نشان داد اعمال فرایند پولیش منجر به تشکیل لایه نازک و پیوسته بر روی سطح می‌گردد.

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