



Evaluation of three kinds of MCrAlY coatings produced by electrospark deposition

Yu-jiang XIE^{1,2}, De WANG², Ming-sheng WANG¹, Wei YE¹

1. State Key Laboratory for Corrosion and Protection, Institute of Metal Research,
Chinese Academy of Sciences, Shenyang 110016, China;

2. Institute of Applied Physics, Jiangxi Academy of Sciences, Nanchang 330029, China

Received 15 July 2015; accepted 6 January 2016

Abstract: Three kinds of MCrAlY coatings with different aluminum contents (5%, 8%, 12%) were prepared by electrospark deposition (ESD), their microstructure and oxidation behavior were studied by SEM and XRD. The results showed that the phase composition of the coatings changed from single γ -Ni to γ -Ni+ β -NiAl hypoeutectic to γ -Ni+ β -NiAl hypereutectic with increasing aluminum content, and their solidification morphologies changed from cluster of cellular structure to cellular dendrites to coarse dendrites with secondary arms. When exposed at 1000 °C, the coatings with higher Al content had a stronger tendency to form θ -Al₂O₃ in the initial several hours. After 100 h of oxidation, a dense and adherent α -Al₂O₃ scale formed on all of the three coatings but the thickness of α -Al₂O₃ scale and the amount of remaining θ -Al₂O₃ increased with increasing Al content. These results revealed different characteristics of ESD MCrAlY coatings which could provide scientific guidance for industrial application.

Key words: MCrAlY; electrospark deposition; microstructure; oxidation behavior

1 Introduction

In recent years, cost effective repair and refurbishment of gas turbine blades have increasingly become an industrial practice because of economic considerations. The coatings applied on the blades will gradually degrade with increasing service time, but this is not uniform due to extremely high surface temperatures at some local areas of the component surface. Usually, the whole coating on the blade surface is stripped and then replaced, which limits the number of allowable blade repairs to 2 or 3 times because of blade wall thinning. Thus, it is highly desirable that a cost effective method can be developed to deposit protective coatings in specific regions in a more convenient way.

MCrAlY-type coatings are amongst the most important protective coating materials applied to counteracting hot corrosion and high temperature oxidation. A variety of methods such as thermal spraying [1,2], laser cladding [3,4] and electrospark deposition [5,6] have been employed to produce MCrAlY coatings. Because the turbine components base

alloys on which the MCrAlY coatings are applied are often thermally sensitive, so that thermal spraying is preferred in industrial application partly because it reduces the thermal damage of the substrate. However, the main drawback of thermal sprayed coatings is their poor mechanical bonding to the base alloy and an additional diffusion heat-treatment is generally required. As an alternative, laser cladding has been proposed to prepare MCrAlY coatings with strong metallurgical bonding, but it may result in hot-cracking when trying to weld the coatings on the thermally sensitive substrate. In contrast, electrospark deposition can be used to produce MCrAlY coatings with high bond strength and minimum thermal damage, especially showing great potential to apply MCrAlY coatings locally. MCrAlY overlay typically contains 17%–30% Cr, 5%–12% Al and less than 1% Y, thus assuring the formation of a protective alumina surface scale upon high temperature services. Some investigations have been made to characterize microstructures and properties of MCrAlY coatings produced by electrospark deposition [6,7], but the knowledge about this is still very limited.

In the present work, three kinds of commercial

MCrAlY powder with different aluminum contents (5%, 8%, 12%) had been prepared by electrospark deposition techniques. The solidification microstructures and oxidation behavior of these coatings were studied.

2 Experimental

A 3H-ES-6 ESD processing installation was employed to deposit the coatings. The deposition was carried out under the conditions of 960 W and 300 Hz using a hand-held gun at room temperature with argon protection.

A Ni-based superalloy, with the composition of Ni–11.5Cr–8.5Co–3.3Al–3.35Ti–1.85Mo–3.7W–3.9Ta–0.05Zr (mass fraction, %), was cut into specimens with dimensions of 10 mm × 15 mm × 2 mm as the substrate material. The surface was ground to 600 grit SiC, cleaned ultrasonically in alcohol, and then blown dry. Three kinds of MCrAlY alloy powder with different aluminum contents (5%, 8%, 12%) presented in Table 1, referred to as MCr5AlY, MCr8AlY and MCr12AlY respectively, were selected for experiments.

Table 1 Chemical compositions of used MCrAlY alloy (mass fraction, %)

Material	Cr	Co	Al	Y	Ta	Ni
MCr5AlY	25	8	5	0.5	–	Bal.
MCr8AlY	20	24	8	0.5	4	Bal.
MCr12AlY	18	22	12	0.5	–	Bal.

The electrode rods were made by laser powder melting/casting forming. The microstructure of the three electrode rods are shown in Figs. 1(a)–(c). In the MCr5AlY electrode materials, the microstructure only consisted of a ductile γ -Ni solid solution matrix, while in the MCr8AlY electrode material, minor β -NiAl phase appeared and dispersed in γ -Ni matrix. In the MCr12AlY electrode material; however, β -NiAl phase had become major phase instead of γ -Ni phase.

The oxidation kinetics of the deposited coatings at the initial oxidation stage had been measured using the thermogravimetry in ambient air at 1000 °C up to 24 h. Additional isothermal oxidation tests without kinetics determination were carried out in ambient air in a muffle furnace at 1000 °C up to 100 h.

The phase compositions of as-deposited and oxidized coatings were investigated by means of X-ray diffractometry (XRD). The morphologies and chemical compositions of the coatings and the oxide scales were observed and determined by scanning electron microscopy (SEM) equipped with an energy dispersive spectrometer (EDS).

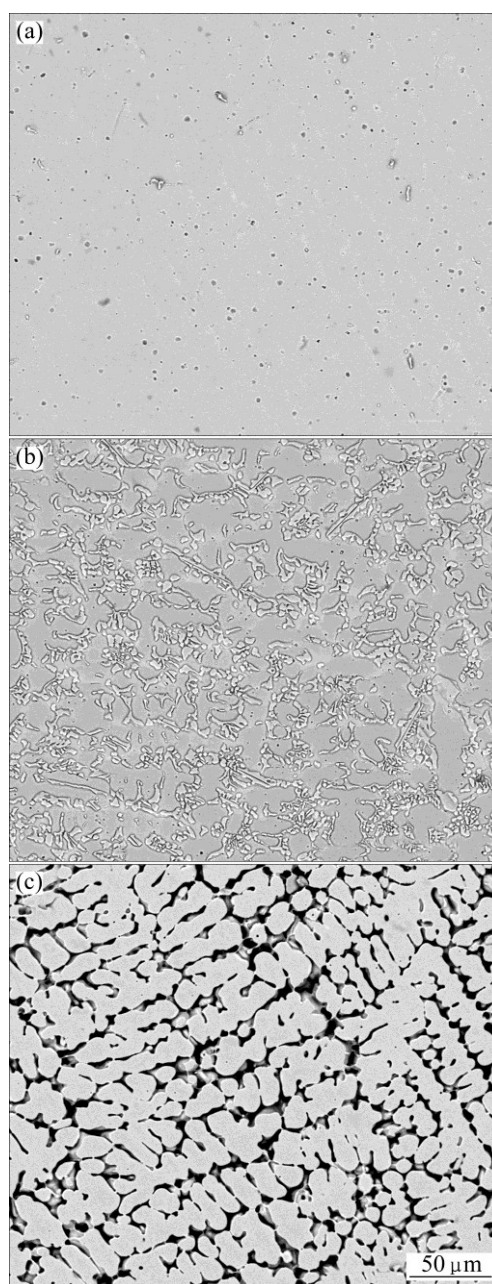


Fig. 1 Microstructure of electrode materials: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

3 Results

3.1 Microstructure of coatings

Figure 2 shows the cross-sectional morphologies of the as-produced coatings. As can be seen, the coatings ranged in 200–300 μ m in thickness. There was apparent diffusion layer lying at the interface between the coating and the substrate for each case, indicating that the coatings were metallurgically bonded to the substrates. The coatings were dense with few pores which could be reduced by selecting appropriate parameters.

Figures 3(a)–(c) show the XRD patterns for the MCr5AlY, MCr8AlY and MCr12AlY coatings

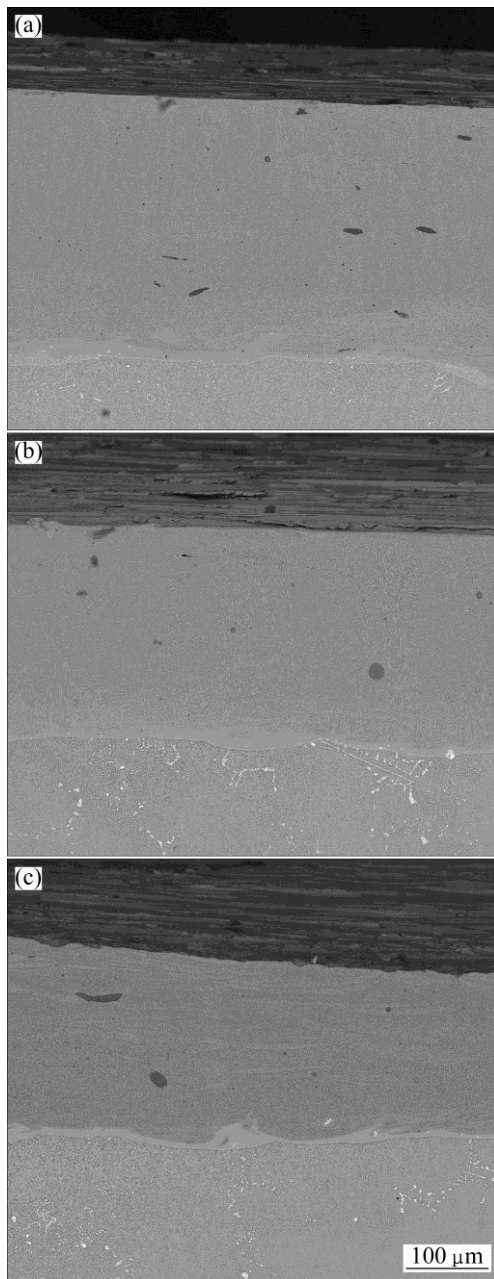


Fig. 2 Cross-sectional morphologies of as-deposited coatings: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

respectively. It can be seen that the MCr5AlY coating consisted of a single γ -Ni phase. With the increase of aluminum content, the deposited coating displayed more diffraction peaks. For the MCr8AlY coating, γ -Ni phase remained as the major phase, but minor β -NiAl phase also appeared (Fig. 3(b)). While for the MCr12AlY coating, more β -NiAl peaks were identified and became the major phase (Fig. 3(c)).

Figures 4(a)–(c) show the microstructures of the MCr5AlY, MCr8AlY and MCr12AlY coatings respectively. All of the coatings were composed of layered thin weld deposits and possessed a columnar structure. However, the columnar structures in the three

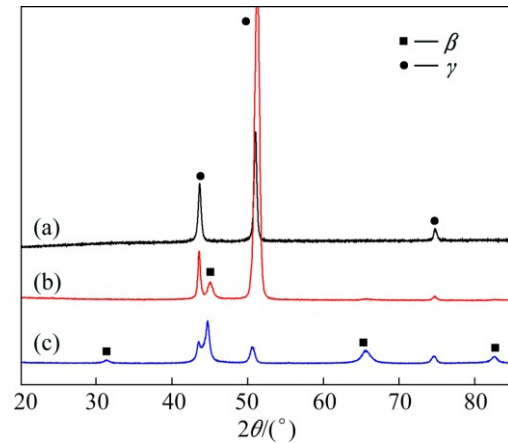


Fig. 3 XRD patterns of deposited coatings: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

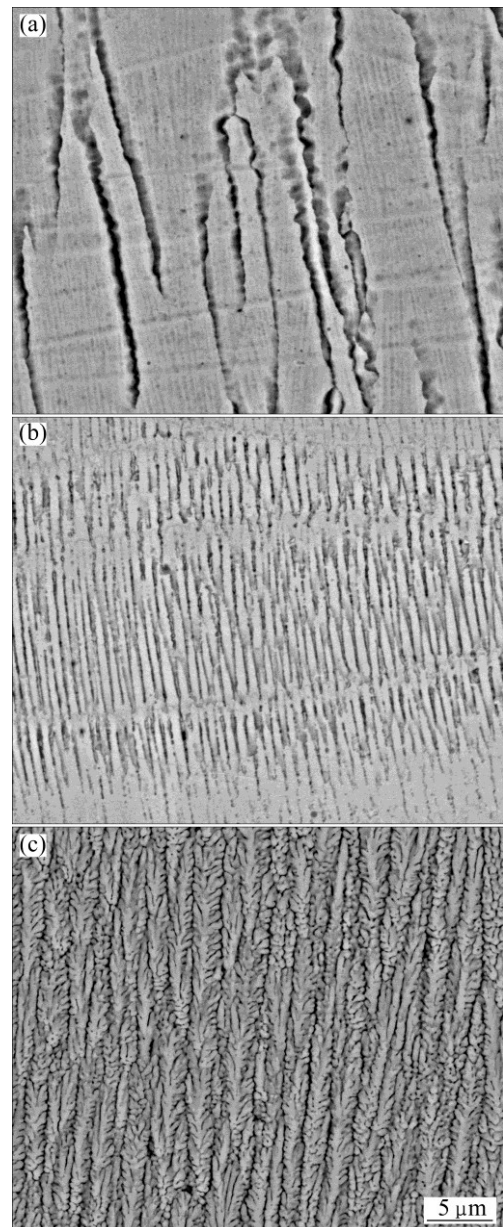


Fig. 4 Microstructures of deposited coatings: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

deposited coatings exhibited some obvious differences. In the MCr5AlY coating, the microstructure consisted of clusters of cellular structure with various growth directions. While in the MCr8AlY coating, the microstructure presented parallel cellular dendrites without secondary dendrite arms. In the MCr12AlY coating, however, secondary dendrite arms grew on the primary dendrite trunks which highly oriented along the growth direction. The primary dendrite arm spacings of the coatings were about 0.3 μm for MCr5AlY, 0.7 μm for MCr8AlY, and 1.7 μm for MCr12AlY, respectively.

3.2 Oxidation behavior

Figure 5 shows the oxidation kinetics curves of the three coatings exposed in air at 1000 $^{\circ}\text{C}$ up to 24 h respectively. The curves were characterized by steeply increase in the initial several hours and then leveling off regardless of the aluminum content in the deposited coatings. In addition, it can be seen that the coatings with higher aluminum content had a greater mass gain than the coatings with lower aluminum content.

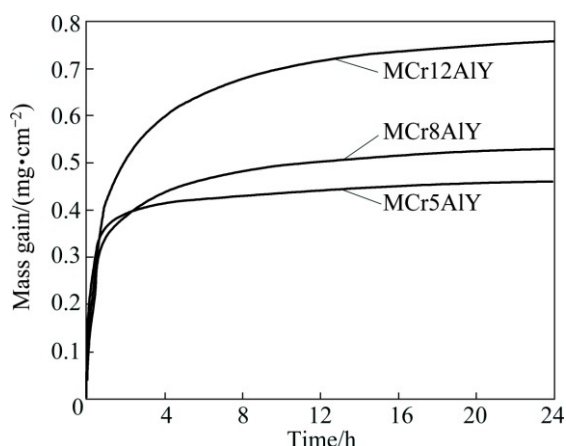


Fig. 5 Oxidation kinetics of deposited coatings at 1000 $^{\circ}\text{C}$ up to 24 h

Figures 6(a)–(c) show the surface morphologies of the oxide scale formed at 1000 $^{\circ}\text{C}$ after 2 h on the MCr5AlY, MCr8AlY and MCr12AlY coatings respectively. For the three coatings, a transient oxide $\theta\text{-Al}_2\text{O}_3$ could be commonly observed at the oxide/gas interface, but the morphologies had a distinct difference with different aluminum contents. For the MCr5AlY coating, a little tiny needle-like $\theta\text{-Al}_2\text{O}_3$ could be observed as independent clusters. While in the case of MCr8AlY coating, a large amount of blade-like $\theta\text{-Al}_2\text{O}_3$ almost covered everywhere. When compared with the previous two coatings, long whisker-like $\theta\text{-Al}_2\text{O}_3$ boomed on the surface of the MCr12AlY coating.

Figures 7(a)–(c) show the X-ray diffraction patterns of the oxides formed on MCr5AlY, MCr8AlY and MCr12AlY coatings after oxidation at 1000 $^{\circ}\text{C}$ for 100 h,

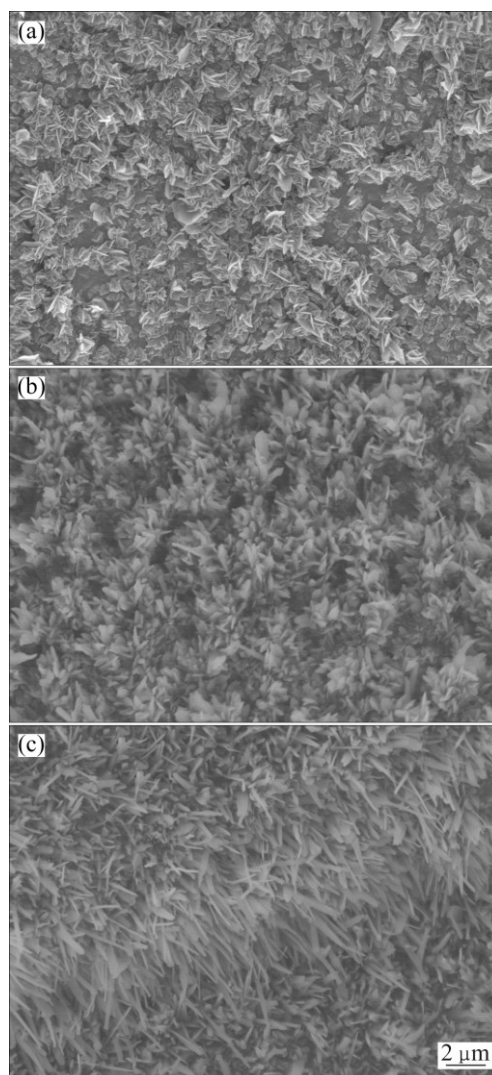


Fig. 6 Surface morphologies of oxide scale formed at 1000 $^{\circ}\text{C}$ after 2 h: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

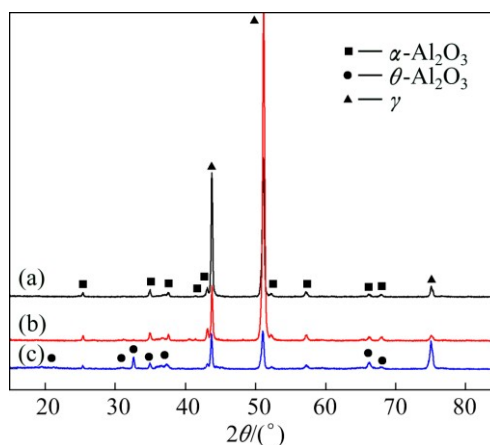


Fig. 7 XRD patterns of deposited coating oxidized after 100 h: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

respectively. For the MCr5AlY coating, only $\alpha\text{-Al}_2\text{O}_3$ peaks were identified. For the MCr8AlY coating, weak $\theta\text{-Al}_2\text{O}_3$ peaks appeared other than $\alpha\text{-Al}_2\text{O}_3$ peaks. As far

as the MCr12AlY is concerned, more and stronger θ -Al₂O₃ peaks were identified in addition to α -Al₂O₃ peaks. The results suggested that the amount of the remaining θ -Al₂O₃ increased with increasing aluminum content in the coatings.

Figures 8(a)–(c) show the surface morphologies of the MCr5AlY, MCr8AlY and MCr12AlY coatings after oxidation at 1000 °C for 100 h, respectively. Different types of surface morphologies were observed on the three coatings. For the MCr5AlY coating, the overall oxide scale was almost covered with flat and short-clubbed α -Al₂O₃ grains. When it comes to the MCr8AlY coating, some needle-like θ -Al₂O₃ was presented as independent colonies and between these θ -Al₂O₃ colonies, some short-clubbed α -Al₂O₃ grains were observable. In contrast to the case of the MCr8AlY

coating, well developed blade-like θ -Al₂O₃ was observed everywhere on the surface of the MCr12AlY coating and because θ -Al₂O₃ formed a complete layer covering on the surface, α -Al₂O₃ formed underneath was hardly seen from surface observation.

Figures 9(a)–(c) show the cross-sectional microstructures of the MCr5AlY, MCr8AlY and MCr12AlY coatings after oxidation at 1000 °C for 100 h. Oxide scale formed on the three coatings was different although all of them had a continuous and adherent layer assumed to be α -Al₂O₃. For the MCr5AlY coating, the oxide scale was pretty simple and thin, only containing a single α -Al₂O₃ layer. With increasing Al content, the oxide scale became thicker and more complex. For the MCr8AlY coating, a few independent oxide grains

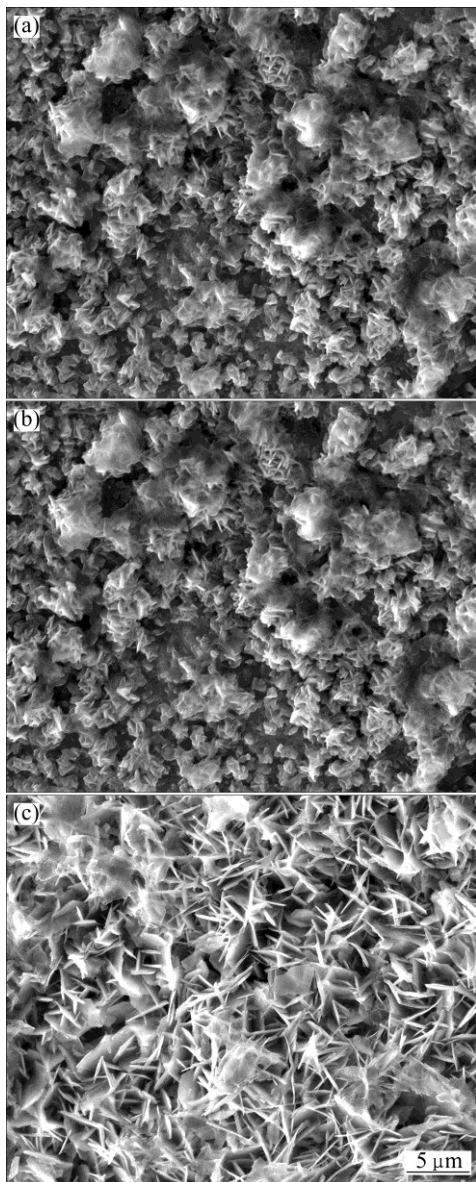


Fig. 8 Surface morphologies of oxide scale formed at 1000 °C after 100 h: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

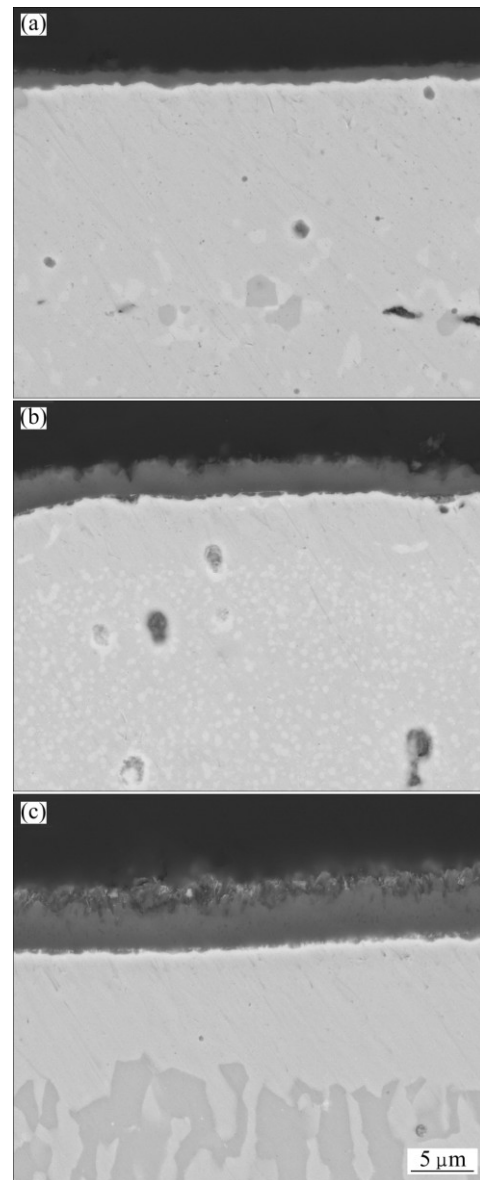


Fig. 9 Cross-sectional morphologies of oxide scale formed at 1000 °C after 100 h: (a) MCr5AlY; (b) MCr8AlY; (c) MCr12AlY

assumed to be θ -Al₂O₃ appeared, adhering to the surface of the continuous α -Al₂O₃ layer. Compared with the MCr5AlY coating and MCr8AlY coating, a complex layered oxide scale formed on the MCr12AlY coating including a discontinuously thick θ -Al₂O₃ outer layer and a continuously dense α -Al₂O₃ inner layer.

4 Discussion

Electrospark deposition is a pulsed micro-welding process and micro-deposit produced by spark discharge can metallurgically bond, even epitaxially solidify to form a dense multilayer coating [5]. During electrospark deposition, individual weld pool is generally around several micrometers to tens of micrometers thick, thus cooling can be considered unidirectional through the thin weld pool and super-high temperature gradient can be expected [6]. With this high cooling rate, over-saturated solid solution state will be obtained, as the dissolved elements have insufficient time to segregate. For MCr5AlY, Al was almost completely dissolved into γ -Ni matrix, and did not segregate under such high cooling rate during such short time, resulting in a single γ -Ni phase in the coating, as can be seen in Figs. 3(a) and 4(a). For MCr8AlY, some residual Al reacted with Ni and forms a trace of β -NiAl, in addition to the major γ -Ni phase, and the high intensity indicated that γ -Ni was highly oriented, as shown in Figs. 3(b) and 4(b). For MCr12AlY, there was enough Al element reacting with Ni to form β -NiAl, and the β phase became the major phase, as shown in Figs. 3(c) and 4(c). However, owing to the high cooling rate, the dendrite has a high tendency to grow directionally along the heat flux direction. Therefore, it is no wonder that the solidification morphologies of the MCrAlY coatings remained columnar dendrites even when the phase compositions changed from single $\gamma \rightarrow (\gamma + \beta \text{ hypoeutectic}) \rightarrow (\beta + \gamma \text{ hypereutectic})$ with increasing aluminum content.

At the same time, small volume of the weld deposit involved and high cooling rate experienced in this process ultimately resulted in a superfine and homogenous microstructure along the coatings. The superfine columnar structure resulted from ESD should serve to enhance the oxidation kinetics by increasing the number of diffusion paths for aluminum along the coating [6,8]. However, the increase of aluminum content might have influenced the cooling rate of the coatings, either by reducing the conductivity of the deposited layers, or reducing the heat generation of the metal couples, which needed further investigation. In fact, the rapid cooling rate can be calculated from the following formula [9]:

$$\dot{T} = (50/\lambda)^3 \quad (1)$$

where $\dot{T}$ is the cooling rate, K/s; λ is primary dendrite

arms spacing, μm . Therefore, the cooling rates for the coatings are 4.6×10^6 K/s, 3.6×10^5 K/s, and 2.7×10^4 K/s, respectively. The solidification velocity can be calculated by

$$v = \dot{T} / G \quad (2)$$

$$G = \Delta T / \Delta x \quad (3)$$

$$\Delta T = T_L - T_0 \quad (4)$$

where v is the solidification velocity, m/s; G is the temperature gradient, K/m; Δx is the average thickness of the deposit, m; T_L is the liquidus, K; and T_0 is the room temperature, K. According to the results and corresponding data in Refs. [3] and [10], the solidification velocity decreased from 7.2×10^{-2} m/s to 7.7×10^{-3} m/s and 5.9×10^{-4} m/s, with the increase of Al content. The higher content of aluminum also changed the composition of the liquid phase at the solidification front, which led to constitutional supercooling [3,11]. All these factors would cause the coarse dendrites with secondary dendrite arms, which would provide more diffusion paths for aluminum and oxygen, as shown in Fig. 4. In addition, the higher content of aluminum and the depletion of β -NiAl phase would provide more Al to diffuse to the surface through these diffusion paths. In summary, with the increase of aluminum content, a faster transport of aluminum to the surface along these high diffusivity paths was possible and formed more transient θ -Al₂O₃, as shown in Figs. 6 and 7. The results are consistent with that obtained by LIU et al [12]. Moreover, the superfine microstructure of the coatings also provided more nucleation sites for alumina [9], this promoted the formation of an alumina scale quickly. These phenomena illustrated why the mass gain of the coating increased steeply with the increasing Al content in the initial stage, as shown in Fig. 5. Generally, Cr₂O₃ and Al₂O₃ will form during high temperature oxidation in MCrAlY coating, and Cr₂O₃ will be preferred when mass ratio Cr/Al > 4:1, while Al₂O₃ forms when Cr/Al < 4:1 [13]. Therefore, Cr₂O₃ might be formed in MCr5AlY coating at the initial stage. However, Cr₂O₃ was not detected by XRD on the surface. This might be caused by the fact that the Cr₂O₃ will change to CrO₃ when oxidized under temperature higher than 900 °C which will volatilize [14]. In other words, the main oxide scale will be Al₂O₃, and it becomes the main concern of the MCrAlY coatings [15,16].

On the other hand, aluminum is a crucial element in MCrAlY alloy system, which may make the phase structure and subsequently the oxidation behavior different. In the present study, the MCr5AlY, MCr8AlY and MCr12AlY coatings consisted of single γ -Ni phase, dual γ -Ni+ β -NiAl phase, dual β -NiAl+ γ -Ni phase respectively. After 100 h of oxidation, the MCr5AlY coating formed a single α -Al₂O₃ scale. In contrast,

MCr8AlY and MCr12AlY coatings formed layered oxide scales, rather than exclusive α -Al₂O₃ scale. The layered oxide consisted of a top θ -Al₂O₃ layer (located at the oxide/gas interface) and a bottom α -Al₂O₃ layer (located at the metal/oxide interface), as shown in Fig. 9. In fact, it has been reported that the phase structure of the coating affected the oxide scale grown on top. Morphologic differences have always been found between the oxide scales grown on β -NiAl and γ -Ni phases: the oxide is thicker and whisker/platelet-shaped on β -NiAl phase, but thinner and smoother on γ -Ni phase [17]. LEVI et al [18] showed initially that both oxides have an inner layer of α -Al₂O₃ and an outer layer (spinel for γ -Ni phase substrate and transient alumina for β -NiAl phase substrate) during high temperature oxidation. The transient alumina (θ -Al₂O₃ and probably also γ -Al₂O₃) on β -NiAl phase substrate might transform to α -Al₂O₃ after further oxidation. However, the transformation from θ -Al₂O₃ to α -Al₂O₃ is generally observed to occur close to or at the metal/oxide interface but not at oxide/gas surface [19]. As described in previous paragraph, more θ -Al₂O₃ scale formed with increasing Al content, the transformation from θ -Al₂O₃ to α -Al₂O₃ initiated at the metal/oxide interface and proceeded outwards. Therefore, the scale might present long whiskers or blades morphology at the oxide/gas interface, indicative of θ -Al₂O₃, but still consisted almost entirely of α -Al₂O₃ beneath this outer layer, and the thickness of α -Al₂O₃ increased, as shown in Figs. 8(c) and 9(c). Due to the formation of a dense α -Al₂O₃ layer, the diffusion of oxygen and aluminum decreased in the oxide, leading to the leveling off of the mass gain curves [20], as shown in Fig. 5.

5 Conclusions

1) Three kinds of MCrAlY coatings with different aluminum contents have been successfully prepared by electrospark deposition techniques. The phase structures of the coatings changed from single $\gamma \rightarrow \gamma + \beta$ hypoeutectic $\rightarrow \beta + \gamma$ hypereutectic with increasing aluminum content. Their solidification morphologies changed from cluster of cellular structure to cellular dendrites to coarse dendrites with secondary arms, due to the decreasing cooling rate and constitutional supercooling.

2) When exposed at 1000 °C, more θ -Al₂O₃ formed with the increase of Al content, which largely increased the growth rate of oxide in the initial stage. After 100 h of oxidation, the coating with the least of aluminum formed a single α -Al₂O₃ scale, the others formed layered oxide including a top θ -Al₂O₃ layer and a bottom α -Al₂O₃ layer. In addition, the thickness of α -Al₂O₃ scale and the amount of remaining θ -Al₂O₃ increased with increasing Al content.

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微弧火花沉积 3 种不同 MCrAlY 涂层的组织与性能评价

谢玉江^{1,2}, 王 德², 王明生¹, 叶 威¹

1. 中国科学院金属研究所, 腐蚀与防护国家重点实验室, 沈阳 110016;

2. 江西省科学院 应用物理研究所, 南昌 330029

摘 要: 应用微弧火花沉积工艺(ESD)制备了 3 种不同 Al 含量(5%, 8%, 12%)的 MCrAlY 涂层, 并采用 SEM 和 XRD 比较研究了显微组织和高温氧化行为。结果表明: 随 Al 含量的增加, 涂层的相组成由 γ -Ni 固溶体变成 γ -Ni+ β -NiAl 亚共晶, 再变成 γ -Ni+ β -NiAl 过共晶, 而相应的凝固组织也由胞状结构团簇变成胞状枝晶再变成带有二次枝晶臂的粗大枝晶。当暴露在 1000 °C 下时, Al 含量越高, 涂层形成 θ -Al₂O₃ 的趋势越明显。当氧化时间达到 100 h 后, 所有涂层表面都生成致密且结合良好的 α -Al₂O₃ 氧化层, 但当 Al 含量升高, α -Al₂O₃ 氧化层厚度和残余 θ -Al₂O₃ 含量都随之增加。

关键词: MCrAlY; 微弧火花沉积; 显微组织; 氧化行为

(Edited by Yun-bin HE)