Steam oxidation behavior of Y-bearing cladding tube with aluminizing coating

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Abstract

A kind of Y-bearing ferritic/martensitic fuel cladding tube with qualified dimensions, acceptable mechanical properties as well as good flattening and flaring performance was manufactured recently by our group. In this paper, to improve and further evaluate its oxidation resistance, the cladding tube was aluminized firstly and then subjected to high temperature steam oxidation at 1200 °C for 8 h. The results indicated that aluminizing coating with gradient content of Al was prepared on the tube successfully. And the matrix microstructure was transformed from tempered martensite into ferrite during aluminizing. Weight gains after high temperature steam oxidation were 72.7 and 1.48 mg cm\(^{-2}\) for the bare and aluminized tubes, respectively. The latter one exhibited better oxidation resistance due to the generated dense aluminum oxide film. Meanwhile, Kirkendall pores were formed near oxidation surface and should be eliminated for the real application of the aluminized tube in the future.

1. Introduction

Fuel cladding is one of the most important safety barrier in fission reactor and zirconium (Zr) alloys are usually selected as fuel claddings in water-cooled nuclear reactors due to the low neutron cross-section, suitable mechanical properties and excellent corrosion resistance [1–3]. However, the reaction between Zr and steam at high temperature is accompanied by the release of large amounts of hydrogen gas [4–6]. According to the reports of the Fukushima Daiichi nuclear disaster, the oxidation of Zr cladding begins above 900 °C and causes accelerated hydrogen generation above 1227 °C [7–9]. For example, during the loss-of-coolant-accident (LOCA), cladding temperature will be increased up to 1200 °C due to the decay heat from fuel accompanied by the evaporation of the cooling water, which results in the rapid steam-oxidation of Zr alloy and further produces significant and dangerous levels of hydrogen [10]. To improve the high-temperature steam oxidation, protective coatings on cladding tubes have been developed recently. Usually, the protective coatings react with steam at high temperature then the dense and continuous Al\(_2\)O\(_3\), Cr\(_2\)O\(_3\) are formed thereby prevent further oxidation of the matrix. For example, Yuan et al [11] investigated the steam oxidation of HR3C austenitic stainless steel in pure steam at 700 °C and found that Cr concentration of 25 wt% in HR3C was sufficient to form Cr\(_2\)O\(_3\) scale, which however was weakened by the generated hydrogen. In [12], Zr alloy with Cr–Al coating was oxidized in steam at 1200 °C and the oxide layers were comprised of Cr\(_2\)O\(_3\) and Al\(_2\)O\(_3\), which resulted in the improved oxidation resistance compared to pure Cr coating. Besides, Wang and Ke et al [13, 14] investigated the steam oxidation of Ti\(_2\)AlN coating at 750 °C and Ti\(_2\)AlC coating at 1000–1200 °C and the results indicated that Al easily diffused to the surface thus formed α-Al\(_2\)O\(_3\). Therefore, Al coatings, Cr coatings and MAX coatings containing Al, Cr elements are promising candidates as oxidation protective coatings for cladding tubes.
Except for the protective coating, matrix material is also another important factor determining the service performance of cladding tube. Ferritic-Martensitic (F/M) steels with body-center-cubic (BCC) structure and 9–12 wt% Cr addition are potential candidates for the cladding tubes due to the low swelling rate and excellent corrosion resistance [15]. Furthermore, compared with the Zr-based alloys, F/M steels exhibit higher thermal conductivity and service temperature. The maximum service temperatures of Zr alloy and F/M steel are 400 and 500 °C based on the neutron irradiation of 10–50 dpa, respectively [2, 16]. However, the main drawbacks of F/M steel serviced as fuel cladding are high neutron cross-section [2], weak high temperature oxidation resistance [17] and microstructural instability under irradiation and high temperature [18, 19], which can be resolved by reducing the thickness of cladding tube [20], aluminizing [21] and oxide dispersion strengthened (ODS) matrix [22], respectively. Thus aluminized ODS F/M steel is being considered as a primary candidate material for fuel cladding in future fission reactor.

Recently, Y-bearing 9Cr-ODS F/M cladding tubes with dimensions of 6 mm outer diameter, 0.5 mm thickness and 3 m length were manufactured successfully by our group and their compositions, microstructure, mechanical properties were reported in [23]: Nominal compositions were 0.08–0.11 wt% C, 8.9–9.5 wt% Cr, 0.3–0.5 wt% Mo, 1.1–1.3 wt% W, 0.45–0.55 wt% Mn, 0.1–0.2 wt% Si, 0.2–0.3 wt% V, 0.15–0.22 wt% Ta, 0.04–0.06 wt% Ti, 0.01–0.2 wt% N, 0.2–0.3 wt% Y. Microstructure was tempered martensite accompanied with 0.6% ferrite and the average size of the prior austenite grain was about 13.4 μm. Besides, the Y-bearing tube exhibited acceptable mechanical properties and met the standards of ultrasonic, flattening, flaring examinations for cladding tube. In the present paper, to improve and further evaluate its oxidation resistance, the cladding tube was aluminized firstly and then subjected to high temperature steam oxidation at 1200 °C for 8 h. Besides, microstructure evolution during aluminizing and oxidation was investigated and the high temperature steam oxidation resistance comparison between the aluminized and bare cladding tubes was also conducted.

2. Materials and methods

Cladding tubes with external diameter of 6 mm and thickness of 0.5 mm were manufactured using Y-bearing 9Cr-ODS F/M steel via the following two steps as shown in figure 1. Firstly, the forged rods with a diameter of 34 mm were subjected to boring, annealing and the initial tubes with the size of Φ32 × 5 mm³ were obtained. Subsequently, the initial tubes suffered cold rolling by 15 passes. To relieve the deformation stress, annealing at 800 °C was performed before each rolling pass. Furthermore, an intermediate heat treatment in terms of quenching at 1050 °C followed by air cooling and tempering at 750 °C followed by air cooling was carried out during cold rolling to improve their deformability. Chemical compositions, microstructure characteristics and mechanical properties of the cladding tubes were presented in [23]. And tube specimens with the size of Φ6 × 0.5 × 10 mm³ were cut from the long cladding tube for the pack cementation aluminizing test. Aluminizing medium was the mixed powders with 45 wt% Fe, 50 wt% Al and 5 wt% impurities. NH₄Cl was selected as the activator. Aluminizing test was conducted at 930 °C for 9 h followed by furnace cooling and the cooling rate was about 0.45 °C min⁻¹. To estimate the oxidation behavior under LOCA, the aluminized cladding tube was subjected to the high temperature steam oxidation. Firstly, specimens were heated to 200 °C with the heating rate of 100 °C min⁻¹, then further heated to 1200 °C with the heating rate of 50 °C min⁻¹. Subsequently, mixed gas with 90% H₂O and 10% Ar was introduced into the oxidation apparatus for 8 h. Ar was selected as the protective gas during the heating and cooling stages.
Microstructure was examined via scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS) detector. SEM specimens were mirror polished and etched via a solution composed of 5% HF and 95% HNO₃. Microhardness was measured by a MH-6 model hardness tester with a load of 250 g and dwell time of 10 s. Nine measurements for microhardness test were carried out to obtain statistically significant data.

3. Results and discussion

3.1. Aluminizing coating

Cross-section microstructure and composition characteristics of the aluminized cladding are shown in figure 2. The aluminizing coating can be divided into four kinds of layers as shown in figure 2(a). Thickness and Al content of each layer can be obtained basing on figure 2. The outermost layer, i.e., layer I exhibited the thickness of ∼100 μm. Al content and Fe content were about 66 at% and 26 at%, respective, thus the atom ratio of Al/Fe was about 2.5, which was well consistent with that of Fe₂Al₅. For the layer II, the thickness was ∼20 μm and Al content was in the range of 45–65 at%. In the case of the layer III, the thickness was ∼50 μm and Al, Fe exhibited the same content was ∼45 at%. The atom ratio of Al/Fe was about 1, which indicated the layer III comprised of FeAl phase. So the layer II was the transition region between Fe₂Al₅ and FeAl. The innermost layer, i.e., layer IV showed the thickness of ∼150 μm and Al content of 0–40 at%. Layer IV was the transition region between FeAl layer and matrix. This transition region can be attributed to the inward diffusion of Al to matrix and the Al content was decreased gradually with increase of distance from outer surface. Due to the Al content was too few to form Fe-Al intermetallic compound, thus layer IV was comprised of Fe matrix and solute Al with gradient distribution characteristic. In brief, the total thickness of the aluminizing coating was about 420 μm, layer I and layer III were comprised of Fe₂Al₅ and FeAl, respectively while layer II was transition region of Fe₂Al₅/FeAl and layer IV was transition region of FeAl/matrix.

Besides, some AlN phases marked by arrows were observed in figure 2(a). Formation of AlN phases should be attributed to the following reasons. Firstly, composition gradient would introduce outward diffusion of N from the substrate together with inward diffusion of Al from surface. Secondly, the packing activator of NH₄Cl would also induce inward diffusion of N in the coatings. Thirdly, AlN exhibits a large negative Gibbs free energy of formation, i.e., −287 kJ mol⁻¹ [24], which facilitates its formation where Al and N are sufficient.

Cross-section microhardness of the aluminizing coating is shown in figure 3. The hardness curve can be divided into four sections, which was similar to the composition curve as shown in figure 2(b). Both curves were comprised of the initial platform, the sharp decline stage, the second platform and the slow decline stage. Thus it can be deduced that the hardness of the layer I, layer II, layer III, layer IV was about 1000, 400–1000, 380 and 170–360 HV, respectively. The two platforms in composition curve and hardness curve indicated that both layer I and layer III were comprised of certain phase components, i.e., Fe₂Al₅ and FeAl, respectively.
3.2. Matrix microstructure after aluminizing

Microstructure of the tube matrix after aluminizing was also examined and presented in figure 4(a). Some chain-like phases were distributed in the matrix, which should be ferrite rather than tempered martensite. In this work, aluminizing was performed at 930 °C for 9 h followed by furnace cooling with the cooling rate was about 0.45 °C min⁻¹. On the one hand, the calculated Ac₁ and Ac₃ were 855 °C, 955 °C, respectively for the steel with similar compositions but with Y element. Therefore, specimens would suffer austenitizing during the aluminizing process. On the other hand, previous literature [25] reported that proeutectoid ferrite would be formed when the cooling rate was lower than 120 °C min⁻¹ after austenitizing for F/M steel. Besides, according to the CCT diagram of the 9Cr-ODS steel [26], when the cooling rate is decreased to 500 °C min⁻¹, only ferrite is formed. Thus the low cooling rate of 0.45 °C min⁻¹ after aluminizing would facilitate the transformation from austenite into ferrite. Furthermore, microhardness of the aluminized matrix was below 200 HV as shown in figure 3. And the microhardness of the matrix before aluminizing (tempered martensite) was about 312 HV. Basing on the above results, it can be concluded that the matrix of aluminized tube was ferrite.

Two kinds of carbides were observed in the matrix of the aluminized tube as marked in figure 4 and the large chain-like carbide was M₂₃C₆ while the small one with the mean size of 0.46 μm was MX basing on the EDS.
results in figures 4(b), (c). Composition of 1# particle was 48.16Cr-44.81Fe-7.03C (wt%). More Cr and C elements were detected thus 1# particle was Cr$_2$C$_6$. Composition of 2# particle was 52.71Fe-28.93Ta-11.48C-5.38Cr-1.50V (wt%). More Ta, C elements as well as some V element were found thus 2# particle was (Ta,V)C. Low cooling rate after aluminizing led to the significant growth of M$_{23}$C$_6$ and finally they were connected with each other thereby exhibited chain-like morphology. Besides, Y&O-bearing particles were also observed in the tube matrix after aluminizing as shown in figure 5(a). It can be seen that some spherical particles with the size of 200 nm were formed on the surface of the central large particle with the size of about 800 nm. EDS results of the central large particle, i.e., 1# particle and the surrounding spherical particle, i.e., 2# particle are presented in figures 5(b) and (c), respectively. Composition of 1# particle was 57.19Fe-19.67Y-8.02Cr-6.50O-6.23Ta-2.39C (wt%). Composition of 2# particle was 63.51Fe-11.44Ta-10.19Cr-5.60Ti-3.42C-3.01Y-2.83O (wt%). More Y, O and less Cr, Ta were detected in the 1# particle while more Ta, Ti, Cr and less Y were detected in the 2# particle. Thus it can be deduced that the central large particle was Y&O-bearing phase and the surrounding spherical particles were carbides. Summarily, microstructure characteristics of the aluminized matrix were ferrite accompanied by chain-like M$_{23}$C$_6$ and ∼0.46 μm MX as well as some Y&O-bearing particles covered by carbides. Briefly, aluminizing coating with gradient content of Al was prepared on the tube successfully and the matrix microstructure was transformed from tempered martensite into ferrite during aluminizing.

3.3. Oxidation kinetics

Usually, oxidation resistance of steels in high temperature steam can be characterized by the weight gain (WG), which can be expressed in equation (1).

$$\text{WG} = \frac{(W_f - W_0)}{S}$$  \hspace{1cm} (1)

where WG is the weight gain in mg cm$^{-2}$, $W_0$ and $W_f$ are the weight of the sample before and after oxidation in mg. $S$ is the superficial area of the sample in cm$^2$. Comparison of WG between the aluminized and bare cladding tubes after high temperature steam oxidation is shown in figure 6(a). For the bare cladding tube, WG increased rapidly with the oxidation time and was kept at about 72.7 mg cm$^{-2}$. Compared with the bare one, the aluminized cladding tube exhibited smaller WG of about 1.48 mg cm$^{-2}$. In other words, the aluminized cladding tube displayed better high temperature steam oxidation resistance than that of the bare one.

Since the test time of 8 h is much shorter than the service time of nuclear reactors, an extrapolation based on fitting available experimental data is needed to provide a reasonable trend in oxide growth with prolonged exposure. The experimental WG data can be fitted using the following equation:

$$\text{WG} = k t^n$$  \hspace{1cm} (2)

where $k$ is a constant, $t$ is the oxidation time in h, and $n$ is an exponent describing the time dependence of the oxide growth. To reveal the oxidation kinetics of the aluminized tube more accurate, the corresponding WG curve is magnified in figure 6(b). It can be seen that the aluminized tube displayed two-stage oxidation characteristics. The first stage was in the range of 0–3.25 h and the second stage was in the range of 3.25–8 h. Table 1 shows the fitting parameters of $k$, $n$ and $R^2$ from equation (2) for the bare and aluminized tubes. The parameter of $R^2$ in the range of 0.92–0.99 indicated that the good reliability of the fitted curves. Besides, it should be noted that the second stage suffered more serious oxidation compared to the first stage for the aluminized
tube according to the oxidation kinetics. Considering the weight gain of the aluminized tube during these two stages was much lower than that of bare tube, it can be deduced that the oxidation difference between the first stage and second stage can be resulted from the aluminizing coating, which will discussed in the following section.

3.4. Post-examination and oxidation mechanism

Surface microstructures of the cladding tubes after oxidation and the corresponding EDS results of the marked regions are shown in figure 7. It can be deduced that the oxidized surface was iron oxide for the bare cladding tube after oxidation basing on figures 7(a), (b). Composition of the marked region in figure 7(a) was 81.28Fe-18.72O (wt%). In the case of the aluminized tube, aluminum oxides were generated as shown in figures 7(c), (d). Composition of the marked region in figure 7(c) was 51.17Al-48.83O (wt%), and the atom ratio of O/Al was about 1.6, which was well consistent with that of Al2O3. Aluminum oxides would inhibit the further oxidation of the matrix and finally resulted in the lower WG as presented in figure 6.

But in the low-magnification SEM of the aluminizing tube after oxidation, some pores were generated as shown in figure 8. As mentioned in 3.1, the aluminizing coating was comprised of Fe2Al5 layer, transition layer, FeAl layer and transition layer from outside to inside. During the initial oxidation process, Al would diffuse outwards from Fe2Al5 layer and react with H2O to form Al2O3 and H2. Meanwhile, some pores were left in Fe2Al5 layer. With the increase of oxidation time, more Al2O3 formed at surface thereby oxidation rate was decreased gradually while more pores were left in the Fe2Al5 layer. On the other hand, H+ would diffuse and trapped in these pores then the expansion of H2 would induce local exfoliation of Al2O3 and the pores were exposed to steam. Once the open pores were formed, inner aluminizing would suffer oxidation significantly as shown the second stage in figure 6(b).

Cross-section microstructure of the bare cladding tube after oxidation is shown in figure 9(a). It can be seen that this cladding tube was oxidized completely and the oxides can be divided into external, transition and internal scales. EDS line in figure 9(b) indicated that main compositions were Fe, O for the external scale while Fe, Cr, O for the internal scale. Thus it can be inferred that the external scale was Fe3O4 and the internal scale was (Fe,Cr)3O4.

Cross-section microstructure of the aluminized cladding tube after oxidation is shown in figure 10(a). Almost no oxidation scale was observed, which was inconsistent with the dense aluminum oxides in figure 7(c).
Thus it can be inferred that the generated aluminum oxide layer was too thin to be detected under this magnification. Besides, EDS line in figure 10(b) demonstrated the uniform distribution of Fe, Al, Cr and O elements from oxidation surface to the tube matrix. Uniform distribution of Al was different from the gradient

Figure 7. Surface microstructures of the bare tube (a), aluminized tube (c) after oxidation and the EDS result of the rectangle region (b) and circle region (d).

Figure 8. Pores formed on the surface of the aluminized tube after steam oxidation.
distribution before oxidation as shown in figure 2(b). Redistribution of Al element during high temperature steam oxidation resulted in the coarsening of ferrite matrix as shown in figure 10(a). Ferrite size was about 242 μm, which was much larger than 21 μm before oxidation in figure 3(a). It is well known that Al is the ferrite favoring element and Al element would diffuse from aluminizing layer to matrix due to the concentration gradient during the high temperature steam oxidation process. Thus both Al element and high temperature facilitated the coarsening of ferrite matrix. In addition, lots of Kirkendall pores formed near the oxidation surface, which can be resulted from the different diffusion rates between Al and Fe in the aluminizing coating. Compared with Fe atom, Al atom exhibits higher affinity with vacancy. Thus more vacancies diffused from the region rich-in Fe to the one rich-in Al thereby resulted in the formation of Kirkendall pores. Besides, dots in ferrite matrix were M23C6 and MX while no Y-bearing particle was observed after oxidation, which can be resulted from the complete covering by carbides.

Summarily, microstructure evolution of the two cladding tubes during high temperature steam oxidation at 1200 °C for 8 h was different as summarized in figure 11. For the bare cladding tube, tempered martensite matrix was fully transformed into Fe3O4 and (Fe,Cr)3O4 scales. In the case of the aluminized cladding tube, ferrite matrix suffered coarsening and the generated dense aluminum oxide film increased the oxidation resistance. However, Kirkendall pores near oxidation surface would limit the real application of the aluminized cladding tube. Thus how to eliminate the Kirkendall pores will be investigated in the future.
4. Conclusions

Recently, Y-bearing F/M fuel claddings with dimensions of 6 mm outer diameter, 0.5 mm thickness and 3 m length were manufactured successfully by our group. Moreover, they exhibited acceptable mechanical properties and met the standards of ultrasonic, flattening, flaring examinations for fuel cladding. Thus in the present paper, to improve and further evaluate its oxidation resistance, the cladding tube was aluminized firstly and then subjected to high temperature steam oxidation at 1200 °C for 8 h. Microstructure evolution of the Y-bearing tube during aluminizing and oxidation was investigated and the oxidation resistance comparison between the aluminized and bare cladding tubes was conducted. The main conclusions were summarized as following:

(1) Aluminizing coating with gradient content of Al was prepared on the tube successfully. And the matrix microstructure was transformed from tempered martensite into ferrite during aluminizing.

(2) After oxidation, WG was 72.7 mg cm$^{-2}$ for the bare tube and the tempered martensite matrix was fully transformed into Fe$_3$O$_4$ and (Fe,Cr)$_3$O$_4$ scales while WG was 1.48 mg cm$^{-2}$ for the aluminized tube and the ferrite was survived but suffered coarsening.

(3) The aluminized tube exhibited better steam oxidation resistance due to the generated dense aluminum oxide film. But the Kirkendall pores near oxidation surface should be eliminated for its real application in the future.

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