Effects of Cr on the interdiffusion between Ce and Fe-Cr alloys

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Abstract

Fuel cladding chemical interaction (FCCI) has been a long-standing issue for the metallic fuel with a steel cladding in a sodium-cooled fast reactor, particularly for a high burnup fuel. Although the FCCI has been largely improved by alloying the fuels with Zr or Pd elements, applying a physical diffusion barrier between fuel and cladding, and employing advanced ferritic/martensitic (F/M) claddings, there is a scientific knowledge gap in understanding the behavior of chromium and its effects on the interdiffusion between lanthanides and advanced F/M steels that contain 9-12 wt. % Cr. In this paper, we systematically studied the interdiffusion between cerium and Fe-Cr model alloys with Cr contents of 6, 9 and 12 wt.%. Following the thermal annealing at 560 °C for up to 100 hours, detailed microstructural characterizations were performed to determine the interdiffusion microstructures, compositional distributions, diffusion kinetics, and phase structures in the interdiffusion zone. This study unambiguously disclosed that, as the Ce diffuses into Fe-Cr model alloys, Cr segregates and precipitates into Cr-rich σ phase consisted of Fe and Cr instead of forming a ternary phase together with Fe and Ce. The precipitation of those nano-sized σ phase particles at the Ce diffusion front would effectively slow down the interdiffusion.

1. Introduction

As summarized by Carmack et al., the metallic fuels for fast reactors have been studied for more than five decades, and with the documented performances, this type of fuel would provide a mechanism for closure of the fuel cycle for reducing the long-lived transuranic isotopes [1]. One of the key performance issues of the metallic fuels is the metallurgical reaction between fuels and their cladding steels, named as fuel-cladding chemical interaction (FCCI) [1-5]. The lanthanide fission products (e.g. cerium) and the actinide fuels (e.g. plutonium) interact with the metal constituents of claddings especially at a high fuel burnup [6]. Iron and nickel in the claddings can form eutectic phases with the outward diffusing lanthanides from the metallic fuels, and those intermetallic phases can have melting points below the operating temperature in a fast reactor [3]. FCCI significantly degrades the integrity of fuel-cladding system and limits the fuel burnup levels. The chemical compatibility between metallic fuel and cladding has been studied by using either in-pile irradiation or out-of-pile high temperature diffusion couple experiments [3-10]. Since then, the FCCI issue has been significantly improved by alloying the fuels with Zr or Pd elements [11], applying a physical diffusion barrier between fuel and cladding, and employing advanced ferritic/martensitic claddings. Chromium, as one of the major alloying elements in advanced cladding steels, has been consistently showing a beneficial effect on improving the FCCI [6, 12]. Cr is not expected to form eutectic phases with lanthanides (or actinides) around the operating temperature between 450C and 650C of a fast reactor [13]. Nevertheless, to date, it is not clear on what the exact behavior or role of Cr is in an interdiffusion process between lanthanides and Fe-Cr binary or Cr containing advanced cladding steels.

Many previous studies mentioned that ternary phases, e.g. Fe-Cr-Ln (Ln: lanthanides) could form during the interdiffusion process. Tortorici and Dayananda performed the diffusion couple experiments using cerium and Fe-10Ni (and Fe-20Cr) at 425°C, and estimated the interdiffusion coefficients using a scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS). Binary

intermetallic phases of Fe_2Ce and Ni_3Ce_7 were identified in the interdiffusion zone. Ternary phases of $(\text{Fe,Ni})_2\text{Ce}$, $(\text{Ni,Fe})_3\text{Ce}_7$, and $(\text{Fe,Cr})_7\text{Ce}_3$ were also claimed to form during the process [6]. Recently, Inagaki and Ogata performed similar diffusion couple experiments using materials of Fe-12Cr and 39Nd-24Ce-13La-12Pr, and they concluded that $(\text{Fe,Cr})_3\text{Ln}$ precipitated in the $(\text{Fe,Cr})_{17}\text{Ln}_2$ matrix at 500-650°C [14]. Another previous study by Kim et al. mentioned that Cr-rich precipitation took place due to the depletion of iron instead of forming intermetallic phases [15]. The chromium concentration appeared to build up in the steel substrate to form a Cr-rich layer, which was also observed during the interdiffusion between U-Zr and Fe-Cr alloy [4, 9, 16]. However, due the fact that previous studies on the lanthanide and Fe-Cr model alloy systems [6, 14], were mainly based on the SEM/EDS analysis or electron probe microanalysis (EPMA), which has limited spatial resolutions for accurately identifying possible sub-micro/nano-sized phases, the exact mechanism of how the Cr in a Fe based alloy interact with the lanthanides and result in less interdiffusion is not clear.

In this paper, the effects of Cr on the interdiffusion between cerium and Fe-Cr model alloys, the system similar to those studied by Tortorici [6] and Inagaki [14], were examined by systematically varying the Cr concentration in model alloys and by using various annealing hours for diffusion kinetics studies. Moreover, advanced characterizations including scanning transmission electron microscope (STEM) and X-ray diffraction (XRD) on specially tailored specimens were applied to elucidate the behavior of Cr segregation and precipitation at a micro/nano scale.

2. Experimental details

The diffusion couple fixture used in this study is shown in Fig. 1. The material of the fixture is Kovar alloy, which exhibits a similar thermal expansion coefficient with those of Fe-Cr alloys and cerium metal. The diffusion couple materials, including cerium and Fe-Cr alloys, were clamped in a sandwich structure between the two bolts. Cerium was selected because of its significant fraction (~22.3 a.t.%) of the total lanthanide fission products (Y, La, Ce, Pr, Nd, Pm, Sm, Eu, and Gd) from a U-Pu-Zr metallic fast reactor fuel pin [17]. Cerium reacts with the cladding constitutions to form intermetallic phases with low melting

points, and it is also one of the major lanthanide fission products that diffuse from fuel into the cladding when a steady state FCCI forms [18]. The cerium sheet (0.8 mm in thickness) was procured from ESPI Metals (Ashland, OR, 97520, US), and the material purity is 3N with other impurity below 500 ppm. The Fe-Cr alloys with nominal chromium contents of 6, 9 and 12 wt.% were fabricated using arc-melting and then were subsequently rolled to 2 mm in thickness at 750°C, and the actual chemical compositions were analyzed by Anderson Laboratory Inc., Greendale, WI, 53129 USA, and they are listed in Table 1. The Fe-Cr model alloys were prepared at the Ames Laboratory (Ames, IA 50011) and the alloy elements have a minimum 99.95% purity. Figure 12 shows the typical grain structure of the Fe-Cr alloys used in this study and the alloys are composed of alpha ferrite.

Prior to the diffusion couple experiments, the Fe-Cr alloys were grinded and polished to a mirror surface finish, and the cerium sheets were only slightly grinded to remove surface oxide using SiC papers without any lubricant and then cleaned using a high-pressure nitrogen gas. Tantalum foils were placed between the diffusion couple and bolts as an oxygen getter and interdiffusion barrier for preventing the reaction between diffusion couple and the fixture material. The loading between the Fe-Cr alloy and cerium was carefully adjusted to 15MPa, which represents a typical contact pressure between the metal fuel and cladding at an immediate level of burnup in a fast reactor []. The diffusion couple was annealed in a tube furnace flowed with a high purity argon gas ($O_2 \leq 1\text{ppb}$) at 560°C for up to 100 hours, and the gas composition is provided by the Airgas, USA, LLC on the website and no other impurity analysis was carried by the authors. The diffusion couples were quickly cooled in air after thermal annealing.

Post annealing, the diffusion couples were sectioned and mounted in an epoxy resin for cross-sectional examinations. Due to the fact that cerium metal is extremely reactive with oxygen and it is nearly impossible to totally eliminate the oxidation during annealing experiment even by using UHP Argon protection gas, the post annealing examinations were all performed at the center of diffusion couples, which would contain a much less oxidation as compared with the areas nearby the edge of diffusion couples based on the visual inspection on the sample surface color. The SEM examinations using

secondary electron and back-scattered electron (BSE) were used to perform an overall survey of the interdiffusion structures, and the SEM/EDS was used for preliminary chemical composition analyses. However, the microanalysis using SEM/EDS has a limited spatial resolution for the features smaller than 1 μ m, the scanning transmission electron microscope (STEM) was employed to further characterize micro/nano-structures and their chemical compositions. The cross-sectional TEM specimens were prepared using a focus ion beam (FIB). The X-ray Diffraction (XRD) was also performed to identify the phases at very near interdiffusion interface that was located by progressively removing the cerium from annealed diffusion couples using a MultiPrepTM system.

3. Results and discussion

3.1 Cross-sectional SEM and EDS analysis

Fig. 3 (a)-(c) shows the cross-sectional SEM images of the diffusion couples between cerium and Fe-Cr alloys annealed for 100 hours. The widths of interdiffusion zone (indicated between the arrows) were measured from multiple locations, and the results are summarized in Fig. 3(d). It can be seen that as the concentration of Cr in Fe-Cr model alloy decreases, the width of interdiffusion zone increases. To analyze the compositional distribution, the EDS line-scans were performed across the interdiffusion zones along the blue lines in Fig. 3(a) and (b). The Fe, Cr and Ce elemental profiles in Fig. 4 indicate that the interdiffusion zone consists of two distinct regions separated by the Matano interface [20] labeled using blue vertical line. In our study, the Matano interface is nearly overlapping with original interface (Kirkendall plane) of diffusion couple since the inward and outward diffusion fluxes are almost same as calculated in the section 3.3 of this paper. Both regions contain Fe and Ce with an atomic ratio of approximately 2 between Fe and Ce, while there is nearly zero concentration of Cr in the cerium part. Moreover, the chromium concentration is found to fluctuate between 5 and 30 at.% in the Fe-12Cr alloy near the Motano interface, which implies that Cr segregates and forms Cr-rich precipitates during the interdiffusion.

The EDS mappings shown in Fig. 5 (b)-(d) were performed at the interdiffusion zone of Fe-12Cr/Ce diffusion couple, and the BSE image of Fig. 5 (a) shows the area of interest. Consistent with the EDS line scan profile shown in Fig. 3(a), iron and cerium inter-diffuse and form a compositional region across the Matano interface. Chromium segregates and forms a localized Cr-rich layer slightly away from the Matano interface into the Fe-12Cr binary alloy, while no Cr diffuses into the Ce. The integral elemental distribution was analyzed by vertically integrating the brightness of pixel of the elemental mappings and the profile is plotted on the top of each image. As shown in Fig. 5 (d), the concentration of Cr largely varies at the interdiffusion direction with a slightly depletion at and near the Matano interface. In addition, Fig. 5 (a) clearly shows a diffusion front as Ce diffuses into Fe-Cr alloy, which is coincidentally reflected by the variation in chromium counts from the EDS line-scan.

The BSE images of Fig. 6(a) and (b) reveal detailed microstructures of interdiffusion zone in the Fe-12Cr alloy, and the dark areas represent low Ce content. Table 2 of SEM-EDS spot analysis shows that the dark areas (the spot 2,4,5) consist of Fe, Cr, and Ce, while the bright areas (spots 1 and 3) contain nearly no Cr and are identified as Fe_2Ce . This analysis clearly shows that the Cr tends to segregate to locations at or near grain boundaries. However, due to the spatial resolution limit of SEM-EDS ($\sim 1\mu\text{m}$), the exact size and chemical compositions of Cr-rich phases cannot be determined, and it is not applicable to conclude whether ternary phases of Fe-Cr-Ce could form in the interdiffusion by solely using the SEM-EDS analysis.

3.2 Composition and phase analyses using STEM and X-ray diffraction

The STEM EDS was used for the 2-D compositional mapping on the Cr-rich precipitates. Fig. 7(a) is a bright-field TEM image of the sample lifted out from the interface between Fe-12Cr substrate and Ce. It is clearly shown that the Cr-rich precipitates can only be observed in the Fe-12Cr side. Fig. 7(b) is a more detailed TEM image of the precipitates and it shows that the normal size of the precipitates is about 200nm. The STEM EDS mapping results in Fig. 7 (c) show that the precipitates consist of only chromium and iron, while the matrix contains iron and cerium with nearly no Cr. The STEM EDS spot analyses in

Fig. 7 (d) also demonstrate that cerium is not present in the Cr-rich precipitates while chromium is not detectable in the matrix. Therefore, it can be concluded that no ternary phases of Fe-Cr-Ce formed during the interdiffusion process.

The XRD results from the interfacial surfaces are shown in Fig 8. The spectrum from the Fe-9Cr/Ce interdiffusion (red line) indicates the presence of Fe_2Ce and a α Fe-Cr substrate, while the spectrum from Fe-12Cr/Ce diffusion couple shows several additional peaks at $2\theta = 46.1, 47.0$ and 76° . Those additional peaks are identified as reflections of $[212]$, $[411]$ and $[532]$ from σ phase. $\text{Fe}_{17}\text{Ce}_2$ was not detected in either Fe-9Cr/Ce or Fe-12Cr/Ce diffusion couple annealed at 560°C in this paper. The identified Cr-rich precipitates have a similar crystal structure to that of σ -phase precipitate in a duplex stainless steel [21, 22] Although the diffraction peaks of Cr-rich precipitates did not appear in the spectrum from the Fe-9Cr/Ce diffusion couple, they were clearly observed in a cross-sectional SEM specimen. As shown in Fig. 9, the mole fraction of σ -phase in a Fe-Cr alloy was simulated using Thermo-Calc by varying the Cr concentration from 20 to 50 wt.%. It indicates that when the chromium concentration increases to 20 wt.%, the proportion of σ -phase becomes significant as temperature approaches 512.7°C or above.

3.3 Interdiffusion kinetics analysis

To study the interdiffusion reaction kinetics, thermal annealing for less than 100 hours were performed. BSE images of Fig. 10 (a) and (b) show the cross-sections of the diffusion couple annealed at 560°C for 12 and 50 hours, respectively. As compared with the 100 hours annealing shown in Fig. 5 (a), the 12 and 50 hours samples clearly display the diffusion initiation path of cerium into the Fe-Cr alloy. The diffusion starts at the grain boundaries of Fe-Cr alloy and then engulfs the grains progressively. The STEM EDS mapping was also performed on a TEM sample lifted out from the diffusion path as indicated (yellow bar) in Fig. 10(a). The STEM image of Fig. 11 (a) shows that cerium diffuses along the Fe-Cr alloy grain boundary and also starts to precipitate into the grains as particle clusters. The EDS mapping of Fig. 11 (b)

further discloses that a Cr-rich shell forms around the cerium particle as the eutectic reaction between Fe and Ce proceeds and it depletes Fe from the bulk surrounding cerium particle.

The interdiffusion fluxes of elements Cr, Fe, and Ce are calculated for Fe-12Cr/Ce and Fe-9Cr/Ce diffusion couples by using the Eq. (1) derived by Dayananda [20]. The calculations are based on the concentration profiles in Fig. 4., and the results are plotted in Fig. 12. The positive value of flux corresponds to the diffusion into Ce (right direction), and the negative value indicates the diffusion into Fe-Cr alloy (left direction). Averagely, the interdiffusion flux $\tilde{J}_{Fe}$ and $\tilde{J}_{Ce}$ are higher in Fe-9Cr/Ce diffusion couple of than those in Fe-12Cr/Ce. This is consistent with the observation that high Cr content in the Fe-Cr alloy can slow down the interdiffusion. For both 9 and 12 Cr model alloys, it is clearly shown that the flux $\tilde{J}_{Cr}$ is closely running against the flux $\tilde{J}_{Ce}$ by following the exactly opposite trend, and this synchronization indicates that the Ce is diffusing against Cr into Fe-Cr alloy. In addition, the flux $\tilde{J}_{Cr}$ at Matano interface is nearly zero for both cases, which implies that Cr does not diffuse across the diffusion couple interface into Ce. Back forward flux and build up Cr was also observed by Totorici and Dayananda in the interdiffusion between Ce and Fe-base alloys [6]. The fluctuation of $\tilde{J}_{Cr}$ becomes evident since at about 10 μm away from the Matano interface X_0 , which corresponds to Cr segregating and clustering. The Matano interface was determined by using Eq. (2) based on the elemental concentration profile of Fe. This observation is consistent with the microstructural characterization results in Fig. 6, where more Cr-rich phases are found at the Ce diffusion front.

$$\tilde{J}_i(X) = \frac{1}{2t} \int_{C_i^-}^{C_i(X)} (X - X_0) dC_i \quad \text{Eq. (1)}$$

$$X_0 = \frac{\int_{C_i^-}^{C_i^+} X_i dC_i}{\int_{C_i^-}^{C_i^+} dC_i} \quad \text{Eq. (2)}$$

$$X_i = \sqrt{2\tilde{D}_i^{eff} t} \quad \text{Eq. (3)}$$

The effective interdiffusion coefficient $\tilde{D}_i^{eff}$ was calculated using the Eq. (3), where X_i is the effective penetration depth of component i in Fe-Cr (L) or in Ce (R) and t is the annealing time. The penetration depth X_i is measured from X_0 to both sides of the interdiffusion zone. The results are summarized in Table 3. Consistently, Ce has a much lower effective interdiffusion coefficient in Fe-12Cr than that in Fe-9Cr and Fe-6Cr, as highlighted in Table 3. The formation of a layer of Cr-rich precipitates shall be the mechanism for slowing down the interdiffusion process. This is consistent with the result reported by Yang [12] that the Cr layer electroplated on HT9 steel has been shown as an excellent diffusion barrier material against FCCI.

Conclusion

The effects of Cr concentration on the interdiffusion between the Ce and Fe-Cr alloys have been fully studied in this paper. The width of interdiffusion zone increases with decreasing Cr content, and it has been shown that Cr can effectively slow down the interdiffusion process for mitigating FCCI in a metallic fuel system. The compositional distributions in the interdiffusion zone have been analyzed using EDS under SEM and STEM. In addition, the intermetallic phases were also determined by using XRD. The interdiffusion zone in the Fe-Cr alloy consists of localized Cr-rich σ phase and Fe_2Ce . Chromium does not form ternary phases together with Fe and Ce, which is consistent with the Ce-Cr phase diagram that no compound will form at the temperature below 1000°C. The interdiffusion kinetics studies show that the diffusion of Ce into Fe-Cr is initiated at the binary alloy grain boundaries and then engulf the grains along with the segregation and precipitation of Cr-rich σ phase from the Fe-Cr matrix. The interdiffusion coefficient analyses quantitatively show that the diffusion rate of Ce in Fe-Cr was nearly reduced into half by increasing the Cr concentration from 9 to 12 wt.%.

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Fig. 1. Diffusion couple fixture and the sample configuration.

Fig. 2. Grain structure of the ferrite Fe-12Cr binary alloy.

Fig. 3. Cross-sectional SEM/BSE images of interdiffusion zones between Ce and: (a) Fe-12Cr, (b) 9Cr, and (c) 6Cr with SE. The BSE image clearly shows the original diffusion interface of Fe-12Cr/Ce diffusion couple, and the widths of interdiffusion zone versus Cr (w.t.%) were summarized in graph (d).

Fig. 4. The EDS line-scan elemental analysis: (a) Fe-12Cr/Ce diffusion couple and (b) Fe-9Cr/Ce diffusion couple. The vertical solid blue lines indicate Matano interface (X_0).

Fig. 5. The BSE images and EDS elemental mappings in terms of Fe, Cr, Ce. The elemental concentration distribution is shown with the averaged brightness profile (or total counts) obtained by integrating the brightness of pixels vertically from each elemental mapping.

Fig. 6. Microstructures of the interdiffusion zone in Fe-12Cr alloy, and (b) is a zoom-in of the area outlined using dash lines in (a).

Table 1

Chemical compositions of 6, 9, and 12Cr model alloys.

Model alloys	Cr (wt.%)	O (wt.%)	Fe
6Cr-Fe	5.96	0.047	Bal.
9Cr-Fe	9.02	0.057	
12Cr-Fe	12.6	0.048	

Table 2

EDS elemental analyses of the spots labeled in Fig. 5(b).

at. %	Fe	Cr	Ce
1	68.0	0	32.0
2	70.3	22.5	7.2
3	68.7	0	31.3
4	52.7	25.5	21.8
5	51.9	30.2	17.9

Fig. 7. (a) Cross-sectional TEM image across the original interface (yellow dash line), and (b) TEM image of precipitates, (c) STEM EDS elemental mapping and (d) EDS compositional analyses on spot 1 and 2.

Fig. 8. X-ray diffraction results from the interdiffusion zone of 9Cr/Ce (red) and 12Cr/Ce (blue).

Fig. 9. Simulated phase fraction versus temperature for Fe-20Cr, Fe-30Cr, Fe-50Cr binary alloys. The σ phase appears at a lower temperature with a higher Cr concentration.

Fig. 10. Cross-sectional BSE images from Fe-12Cr/Ce with reduced annealing time (a) 50 hrs. (b) 12 hrs. (c) Zoomed-in of (b) showing Cr accumulation at interface.

Fig. 11. (a) Bright-field STEM image from Fe-12Cr/Ce after 12hrs of annealing. (b) STEM EDS element mapping on selected area (dashed line).

Fig. 12. The calculated diffusion flux $\tilde{J}_i(X)$ of Fe, Cr, Ce from the diffusion couples of (a) Ce vs. Fe-12Cr and (b) Fe-9Cr vs. Ce, corresponding to Fig. 3(a) and (b), respectively. The arrows indicate the direction of diffusion.

Table 3

The effective interdiffusion coefficients and the effective penetration depths for the diffusion couples of Ce vs Fe-12Cr, Fe-9Cr and Fe-6Cr model alloys. The subscript *L* indicates the interdiffusion in Fe-Cr; the *R* for the interdiffusion in Ce.

Diffusion couple assembly	$\tilde{D}_{i,L}^{eff}$ (m ² /sec)	$\tilde{D}_{i,R}^{eff}$ (m ² /sec)	$X_{i,L}$ (μm)	$X_{i,R}$ (μm)
Fe-12Cr vs. Ce	$\tilde{D}_{Fe,L}^{eff}=7.8\times10^{-16}$	$\tilde{D}_{Fe,R}^{eff}=3.0\times10^{-16}$	$X_{Fe,L}=23.8$	$X_{Fe,R}=14.7$
	$\tilde{D}_{Ce,L}^{eff}=6.8\times10^{-16}$	$\tilde{D}_{Ce,R}^{eff}=2.5\times10^{-16}$	$X_{Ce,L}=22.1$	$X_{Ce,R}=13.5$
Fe-9Cr vs. Ce	$\tilde{D}_{Fe,L}^{eff}=1.6\times10^{-15}$	$\tilde{D}_{Fe,R}^{eff}=5.1\times10^{-16}$	$X_{Fe,L}=34.2$	$X_{Fe,R}=19.1$
	$\tilde{D}_{Ce,L}^{eff}=1.3\times10^{-15}$	$\tilde{D}_{Ce,R}^{eff}=4.7\times10^{-16}$	$X_{Ce,L}=30.3$	$X_{Ce,R}=18.4$
Fe-6Cr vs. Ce	$\tilde{D}_{Fe,L}^{eff}=5.0\times10^{-15}$	$\tilde{D}_{Fe,R}^{eff}=1.8\times10^{-15}$	$X_{Fe,L}=60.0$	$X_{Fe,R}=36.5$
	$\tilde{D}_{Ce,L}^{eff}=5.0\times10^{-15}$	$\tilde{D}_{Ce,R}^{eff}=1.8\times10^{-15}$	$X_{Ce,L}=60.0$	$X_{Ce,R}=36.0$