Oxidation resistance of Fe20Cr2Si alloy in 1200°C steam environment

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1. Introduction

In order to prevent the severe accident of nuclear power plants, many ATF (Accident-tolerant fuel) cladding concepts have been proposed in the world, such as coated Zr alloy, FeCrAl, SiC, Cr-Mo alloy etc. [1-2]. The MMLC (Multi-metallic layered composite) cladding is one of the ATF cladding concepts. It is mainly composed of Zr alloy, FeCrSi alloy, and buffer materials in between them. Among these materials, the FeCrSi alloy is responsible for oxidation protection. In the previous study [3], the Fe20Cr2Si alloy is showed excellent oxidation resistance in 100 sec high temperature steam oxidation testing. However, in order to apply to actual fuel cladding, it needs to be verified that the MMLC cladding can withstand in high temperature steam for longer time, at least a couple of hours. In this study, we conducted long-term high temperature steam oxidation test of Fe20Cr2Si alloy in order to verify the oxidation resistance of MMLC cladding.

2. Experimental Methods

The Fe20Cr2Si alloy specimens of 30 × 10 × 2 mm coupons were prepared for the evaluation of oxidation resistance with time. Before the oxidation test, these specimens were polished using up to 3 micrometer diamond suspension and cleaned with ethanol, acetone, and pure water in series. The steam oxidation experiments were conducted at 1200 ℃ in a tube furnace, where steam and Ar gas of 500 cc/hr is supplied, referring to NRC Regulatory Guide DG-1262 [4]. The steam and Ar gas were supplied after heated to 350 ℃ in order to prevent the generation of condensation water. After the high temperature furnace was stabilized at 1200 ℃, the specimen was inserted into the furnace. The oxidation tests were conducted in 100 sec, 10 min, 1 hr, and 3 hr. The schematic of the oxidation tube furnace is shown in Fig. 1.

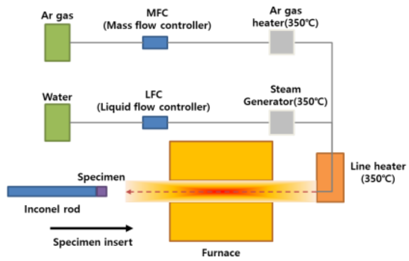


Fig. 1. Schematic of high temperature steam oxidation furnace

3. Results and Discussion

In the previous study [3], we conducted high temperature steam oxidation test of FeCrSi alloys with Cr contents at 1200 ℃ for about 100 sec. In the test result, it is observed that the high temperature oxidation resistance of Fe12Cr2Si alloy and Fe16Cr2Si alloy is not as good as Zr-Nb-Sn alloy. However, it is observed Fe20Cr2Si alloy has excellent high temperature oxidation resistance. The results are shown in Figs 2. and 3.

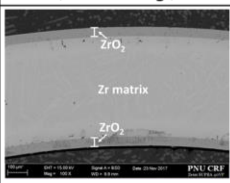
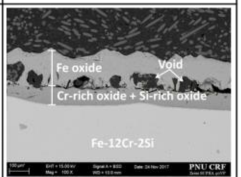
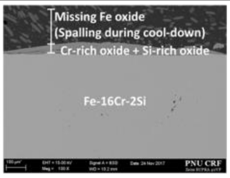
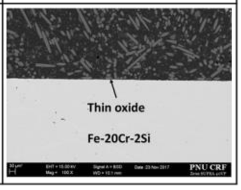
Specimen	Ref. Zr-Nb-Sn alloy	Fe12Cr2Si
Weight-gain (mg/cm^2)	10.07 (3 times avg.)	15.71
SEM (x100)		
Specimen	**Fe16Cr2Si**	**Fe20Cr2Si**
Weight-gain (mg/cm^2)	-29.60 (Missing outside oxide)	0.23
SEM (x100)		

Fig. 2. Comparison of oxidation resistance of Ref. Zr-Nb-Sn alloy and FeCrSi alloys [3]

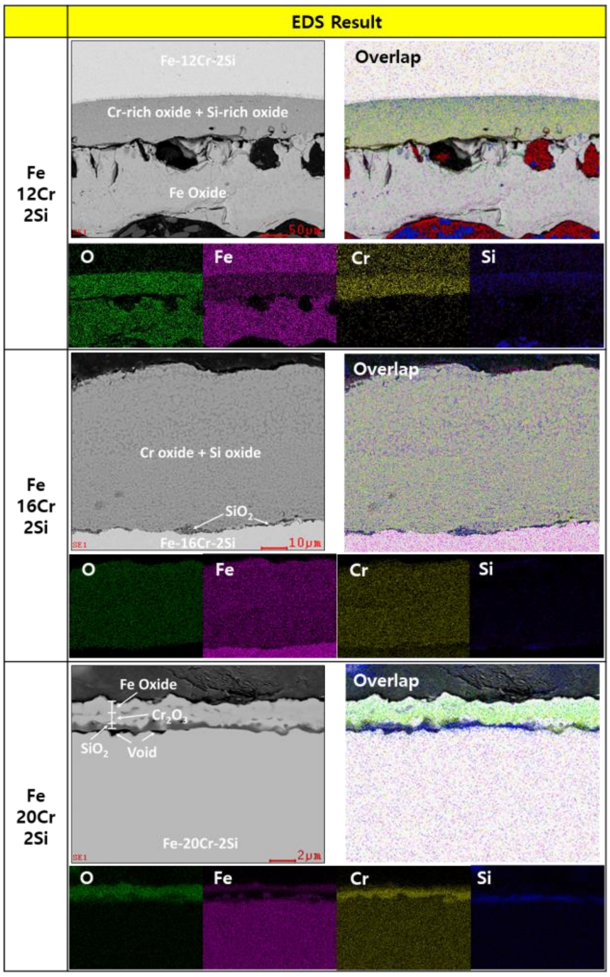
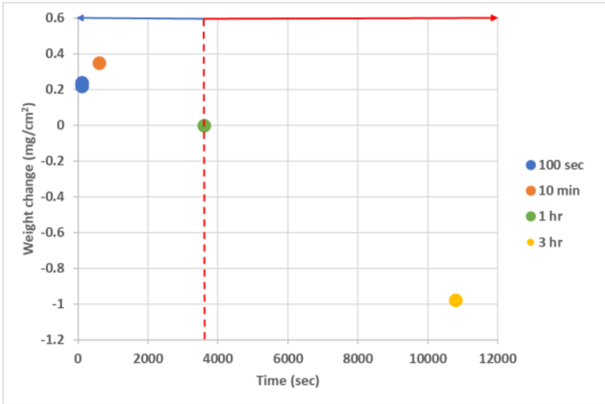


Fig. 3. EDS results of FeCrSi alloys [3]

In order to evaluate the oxidation resistance of Fe20Cr2Si with times, the analyses of weight change and oxide layers were conducted using SEM (Scanning electron microscope) and EDS (Energy-dispersive X-ray spectroscopy) mapping after the high temperature steam oxidation tests.

3.1. Weight change and surface observation

Before the analysis of SEM and EDS, the analysis of weight change was conducted. The results are shown in Fig. 4.



	100 sec	10 min	1 hr	3 hr
1	0.23	0.35	0 (oxide spalling)	- 0.98 (oxide spalling)
2	0.22	-	-	-
3	0.24	-	-	-

Fig. 4. Weight change of Fe20Cr2Si alloy with time

As shown in Fig. 4, the oxidation resistance of Fe20Cr2Si alloy is very high up to the 1 hr test, which is consistent with of the previous study [3]. However, this alloy does not have good oxidation resistance when the exposure time becomes longer than 1 hr. In less than 1 hr tests, a slight weight gain is shown. However, the weight loss is shown in more than 1 hr tests. It is also observed that the outer oxide layers are spalling out in more than 1 hr tests, and some white area is observed. The surfaces of specimens after the oxidation testing are shown in Fig 5.

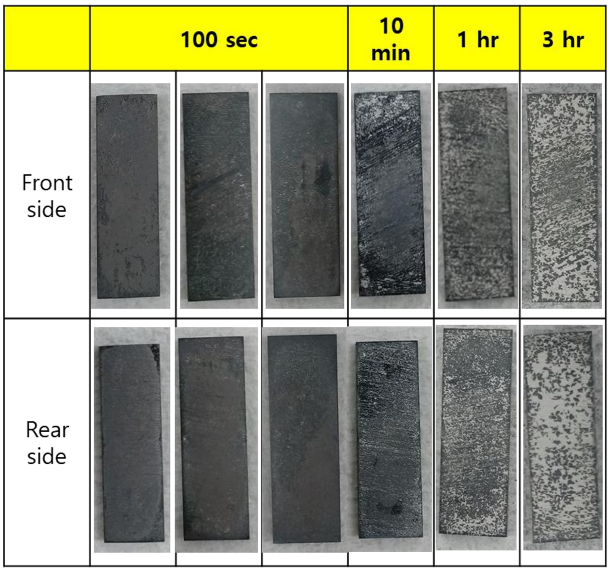


Fig. 5. Surface images of Fe20Cr2Si after the high temperature steam oxidation testing

3.2. SEM and EDS results

In order to observe the oxide thickness and composition, the analysis of SEM and EDS was conducted. The results are shown in Figs. 6 and 7.

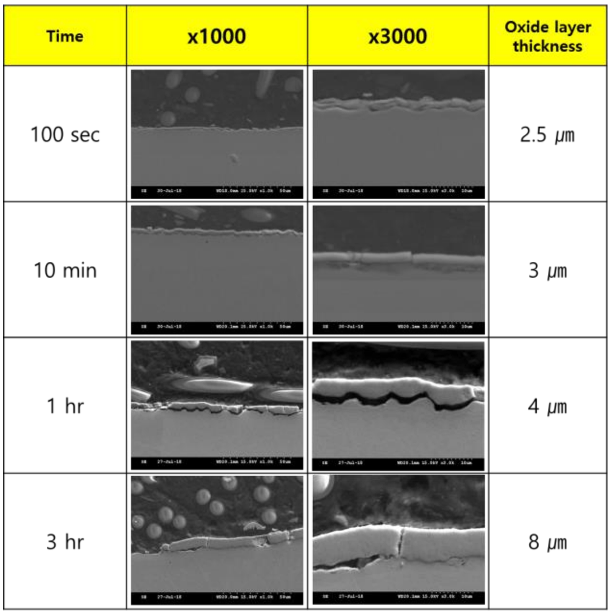


Fig. 6. SEM cross-sectional images of the oxides formed on Fe20Cr2Si after the oxidation testing

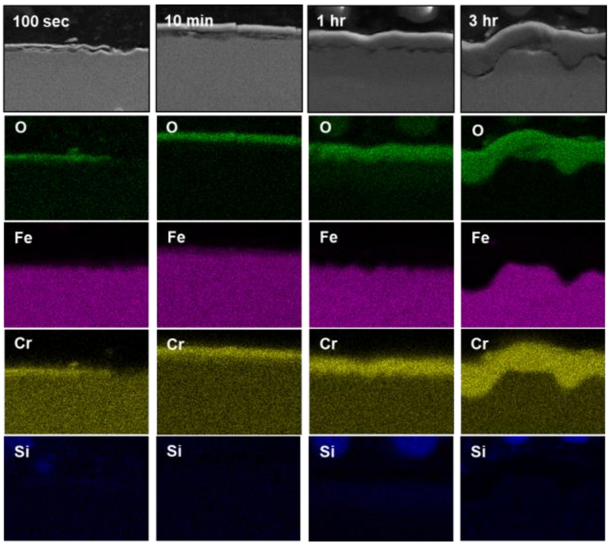


Fig. 7. EDS mapping results for the cross-sectional oxide images for Fe20Cr2Si after the oxidation testing

It is observed that the thickness of oxide layers increases with increasing oxidation time as shown in Fig. 6. The oxide thickness increases to about 8 μm in the 3 hr test. In Fig. 7, it is observed that the Cr oxide layer appears to be mainly formed. In the previous study [3], the Si oxide layer was observed at the metal-oxide interface in the 100 sec test. However, in this study, the Si oxide layer is not observed in this EDS analysis. In order to determine the detail composition of oxide layers, the additional analysis needs to be conducted, especially at the higher magnification.

4. Conclusions and Future Work

In this study, the oxidation resistance of Fe20Cr2Si alloy is evaluated with time in 1200 ℃ steam environment. The oxidation resistance of Fe20Cr2Si alloy is excellent in less than 1 hr tests. However, it starts to show the weight loss due to oxide spalling in more than 1 hr tests. In addition, it is observed that the thickness of Cr oxide layer increases with increasing the steam exposure time.

Additional analysis using SEM, XRD, TEM etc. needs to be conducted, focusing on the behavior of Cr oxide and Si oxide layers with increasing the exposure time. In a long-them steam oxidation test, it can be observed if the Cr oxide layer is volatile and the Si oxide layer is dissolved out.

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