PROGRESS ON THE CORROSION SCREENING TESTS AND MATERIALS SELECTION FOR SUPERCRITICAL WATER COOLED REACTORS

Lefu ZHANG[1], Yichen BAO[1], Li LI[1] and Rui TANG[2]
[1] School of Nuclear Science and Engineering, Shanghai Jiao Tong University
No. 800 Dongchuan Road, Shanghai, P. R. China, 200240
[2] National Key Laboratory for Nuclear Fuel and Materials, Nuclear Power Institute of China
P.O.Box 436, Chengdu, Sichuan, PR China, 610041

Abstract

For a pressure vessel type SCWR concept with an outlet temperature of 510°C, the hot spot temperature on fuel the cladding exceeds 600°C at normal operation conditions, and will be much higher during transients. Selection of fuel cladding material is one of the difficulties due to the high corrosion rate of most alloys in supercritical water. Corrosion screening tests have been conducted for more than 3 years on SCWR candidate materials, both commercially available materials and experimental ODS steels. Literature review and corrosion tests showed that the most probable fuel cladding material may have an austenitic structure and contain high Cr concentration up to 22% or higher, such as HR3C and 310. Aluminium is also helpful to improve corrosion resistance in SCW. The excellent performance in corrosion tests shows that alumina forming, high Cr concentration ODS steels are also a possibility for use as SCWR fuel cladding material, assuming the ductility and formability can be increased.

1. Introduction

Among in-core structural components of a supercritical water-cooled reactor (SCWR), the fuel cladding faces the most severe working conditions. The temperature of hot spot on the fuel cladding of both current pressure vessel and pressure tube conceptual designs exceeds 600°C at normal operations [1]-[4], and will be much higher during transients. Zirconium alloys used for nuclear fuel cladding of current pressurized water reactors (PWR) and boiling water reactors (BWR) are not suitable for use in SCWR due to their high corrosion rate and loss of strength at temperatures higher than 360°C. Other in core structural components work in similar or less severe conditions as the fuel cladding, and thus can be made from similar material as fuel cladding. Therefore, developing the fuel cladding materials is presently one of the most important R&D work for the SCWR.

2. Considerations in Selection of the Candidate Materials for SCWR Fuel Cladding

2.1 Service Conditions of Fuel Cladding

The major safety functions of the fuel cladding are to contain nuclear fuel and retain radioactive fission products during various operating conditions of a reactor. Maintaining the integrity of cladding is of the first importance. For a typical pressure vessel type SCWR concept design as proposed by Liu, Yang and Cheng [3], the outlet coolant temperature is about 510°C and the pressure is about 25MPa, and the maximum normal working temperature of the fuel cladding is up to about 610°C, and will reach above 750°C at transient and accident conditions. The high temperature and pressure water environment, the rather small wall thickness (0.5~0.6mm), the high neutron flux and the long term service require the fuel cladding materials to have high yield and creep strength sufficient to resist the normal outside pressure of about 25MPa at temperatures up to 750°C, good corrosion resistance to both water and fission products to avoid thinning by general corrosion and growth of stress corrosion cracks, and low susceptiblity to irradiation embrittlement, swelling and irradiation induced accelerated corrosion.

2.2 Estimation of Stress on Cladding Tube Wall

Fuel rods of water reactors are generally pressurized by helium gas to increase the rigidness of the cladding tube, to balance the outside compression pressure applied by the high pressure reactor coolant and to improve the thermal conductivity of the gap between fuel pellets and the cladding inner wall. Initial pressurization of PWR fuel rods is 2 to 3 MPa, which may develop about 6 MPa internal pressure during normal working conditions. However, a loss of coolant accident (LOCA) will cause the loss of primary coolant pressure, and the internal pressure of the fuel rod will apply a tensile stress on the cladding tube wall, which is dangerous and probably leads to cladding tube rupture. For a typical SCWR design [3] with an outlet temperature of 510°C and an operating pressure of 25MPa, the net compression pressure at normal operation condition and internal pressure applied on cladding tube during a LOCA are diagrammatically shown in Figure 1, with different initial helium pressurization.

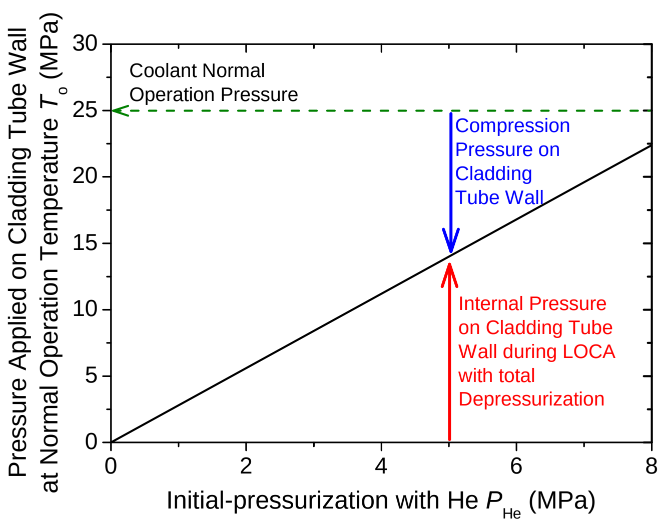


Figure 1 Outside and internal pressure applied on fuel cladding during normal and LOCA conditions with different initial pressurization.

Let us make a rough estimation of the stress on the cladding tube. For the SCWR concept design in reference [3] with a cladding outer diameter of 8mm and tube wall thickness of about 0.6mm, the stress loaded on the tube wall by water of 25MPa is about 160MPa if no initial pressurization exists inside the fuel rod. The stress of 160MPa is rather high compared to the yield strength and creep strength of commonly used stainless steels, such as 316L at temperatures over 550°C.

Supposing that the fuel rods are initially pressurized by helium to 2, 3, 4, 5 and 6 MPa at room temperature, the internal pressure developed are about 6, 9, 12, 15 and 18 MPa at normal fuel rod working temperature of 650°C, respectively, and the differential compression stresses loaded by the SCW coolant at 25MPa are about 126, 105, 85, 64 and 44MPa, respectively. Internal pressurization by helium significantly reduces the differential stress applied on the cladding tube.

However, during a LOCA in which the reactor coolant is depressurized, the fuel cladding tubes need to withstand the stresses developed by the internal pressure of helium, which may be from 41 up to 120 MPa for initial helium pressure of 2 to 6MPa. To make a compromise, the initial pressure of helium in the cladding tube can be kept to about 4MPa, and the stress on the tube wall can be reduced to about 85MPa both at normal working conditions and total depressurization conditions caused by a LOCA, assuming that the maximum fuel rod temperature is kept below 650°C. If a LOCA causes fully loss of reactor pressure and overheating of fuel brings the cladding temperature up to 800°C, the stress developed on the tube wall can still be kept to about 95MPa with the fuel rod initial helium pressure of 4MPa.

2.3 Loss of Strength due to Corrosion

The inlet coolant water, which is in the subcritical state at a temperature of about 280°C, flows through the core and is heated up to the supercritical state. The corrosion behaviours of metallic materials, such as the commonly used iron based and nickel based PWR materials, are different in subcritical, near critical and supercritical water. In the subcritical region, besides oxidation, electrochemistry controlled corrosion such as dissolution and deposition, intergranular attack, and stress corrosion cracking (SCC) are the dominant corrosion mechanisms. The corrosion rate of stainless steel and nickel base alloys in subcritcal water is generally determined by the SCC crack growth rate. Supercritical water is a very oxidizing medium, and oxidation is the dominant corrosion mechanism. The corrosion rate in SCW is determined by the diffusion of oxygen atoms into base metal and alloying atoms out to the surface. The high temperature provides the activation energy for diffusion of atoms in the material. High temperature and pressure cause the dissociation of water molecules and provides a source of dissolved oxygen [15] whose concentration can drive the formation of high valence metallic oxide, such as Fe_2O_3.

SCC and oxidation both cause a loss of wall thickness, and therefore cladding strength loss. The thickness of SCWR fuel cladding is generally 0.5~0.6mm. Loss of strength or rigidity will lead to crashing or deformation of the cladding tube under high operating pressure. Total thinning by general corrosion, penetration of stress corrosion cracks and intergranular attacks should in no case exceed 5% during its 3 to 4 fuel cycles in SCWR. Thinning of the cladding tube wall by 5% will increase the tube wall stress by 10%, which means about 10MPa higher stress will be applied on the cladding tube wall, and this will significantly reduce the safety margin of the fuel rod.

2.4 Swelling, Embrittlement and Loss of Strength due to Irradiation Damage

Irradiation damage of cladding materials is another important issue. The irradiation conditions of SCWR fuel cladding will be more similar to the case of a sodium cooled fast breeder reactors (FBR), but the fast neutron fluence is comparatively 2 to 3 factors lower.

Much research experiences and data about radiation damage of FBR fuel cladding materials can be used for reference in the selection of SCWR cladding materials.

Austenitic stainless steels, ferritic/martensitic steels and oxide dispersion strengthened steels have been intensely investigated with respect to radiation damage [12]-[13]. Based on current knowledge, it is known that irradiation swelling of ferritic/martensitic steels is about two to four times lower than austenitic stainless steels, cold-working usually decreases void swelling of ternary Fe-Cr-Ni alloys at relatively low irradiation temperatures, but generally increases swelling at higher irradiation temperatures. The fast neutron fluence of SCWR fuel cladding is less or near to the fluence of the onset of swelling, and therefore irradiation swelling will not be a critical concern. But irradiation damage induces segregation of alloying elements at grain boundaries, and hence will induced stress corrosion cracking and/or intergranular attack in supercritical water [14], which should arouse great concern by the designers.

2.5 General Technical Requirements of SCWR Fuel Cladding

High temperature strength, general corrosion rate, and radiation induced stress corrosion cracking would be of great concern in the selection of materials for fuel cladding. According to the analysis described above, the major requirements of the candidate cladding materials for SCWR are as follows:

(1) Minimum yield strength at 650 and 800°C should be 150MPa and 100MPa, respectively;
(2) Minimum creep strength at 650 and 800°C should be 120MPa and 90MPa, respectively;
(3) Low general corrosion and low susceptibility to stress corrosion cracking and intergranular attack, and total thinning must be in no case exceed 25μm during the 3 to 4 fuel cycles;
(4) Very low or no susceptibility to irradiation induced segregation at grain boundaries, and hence unsusceptible to radiation induced stress corrosion cracking;
(5) Low swelling and unsusceptible to embrittlement by neutron irradiation.

3. Candidate Materials for SCWR Fuel Cladding

Zirconium alloys have been used as the cladding materials for water cooled reactors for more than 50 years, and have been considered the best suitable material for the operation conditions water cooled reactors. However, zirconium becomes reactive in water at temperatures above 360°C, and the mechanical strength falls rapidly when the temperature is increased over 400°C. General corrosion tests of various zircaloys were conducted in SCW up to 500°C, and results showed high weight gain rate [7], and the possibility for their use in SCWR is very low.

Presently available materials for the fire tubes of ultra-supercritical fossil plants, piping materials used in high temperature and pressure chemical systems, and materials for aero-engine blades, can meet the high temperature mechanical requirements, while the nuclear properties and degradation behaviours under neutron irradiation should be evaluated. Fuel cladding materials used in sodium-cooled fast breeding reactors, and materials for fusion reactor system usually have excellent properties against irradiation damage, however their mechanical strength and corrosion resistance in SCW need to be evaluated. These materials partly meet the technical requirements of SCWR cladding, and can find potential application in SCWR. Ferritic/Martensitic steels such as T92, HCM12A and HT-9, austenitic stainless steels such as 304NG, AL-6XN, 310 and 800H, nickel base alloys, such as alloy 690, 625, 718 and C276, ODS steels such as MA956, 9~18Cr-ODS steels, have been evaluated researchers during the past years. The review article by Sun [5] gives a general comparison of most of the candidate materials.

Corrosion screening tests of candidate materials in supercritical water is one of the most important research topics at present. Based on the current available results [16], most of the short-listed materials show high oxidization rates, especially when the temperature is above 600°C. Only high Cr content austenitic stainless steels, nickel base alloys, and alumina film forming alloys can meet the stringent corrosion requirement.

4. Progress on the Corrosion Screening Tests

4.1 Corrosion Screening Tests

Corrosion screening tests of candidate materials (as shown in Table 1) were conducted in our supercritical water autoclave both in static and circulating water mode. Testing temperatures were 550, 600 and 650°C, and the pressure was kept at about 25MPa. Weight gains of the materials are shown in Figure 2, and a comparison of the corrosion rate between each material is given in Figure 3.

The candidate materials tested covers ferritic/martensitic steels, austenitic stainless steels and nickel base alloys. The Cr concentration of ferritic/martensitic steels varies from 9 to 18%, including F92, X20Cr, 410, 14Cr-ODS, 18Cr-ODS and MA956, in which ODS steels are also included. Two types of austenitic stainless steels were tested, 304NG and AL-6XN. The nickel base alloy, Hastelloy C-276, was tested as a reference.

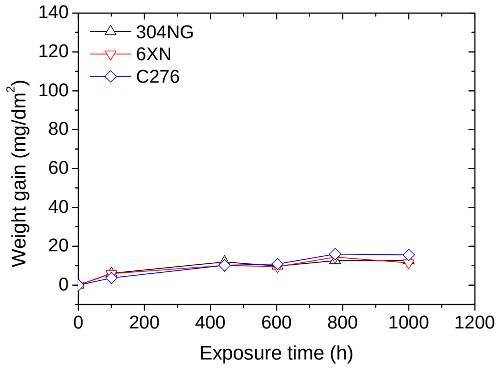
4.2 Corrosion Behaviour of Austenitic Stainless Steels

Austenitic stainless steels have been successfully applied in LWR and FBR internals, and are considered to be high priority candidate material for the SCWR. Austenitic stainless steels exhibit very low oxidation rate in SCW at temperatures lower than 550°C, as shown in Figure 2a. Weight gain rate is comparable to the nickel base alloy Hastelloy C276. However, with the increase of SCW temperature, especially up to above 600°C, large differences in corrosion rate can be observed from different stainless steels, as shown in Figure 2b, c and d.

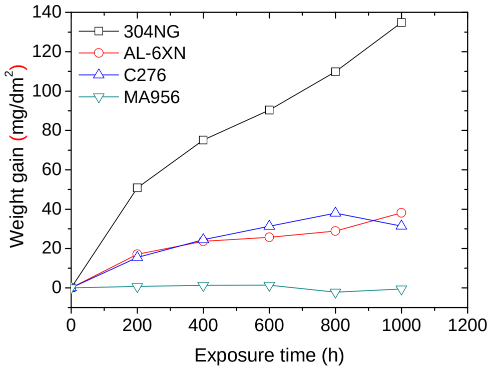
Higher concentrations of Cr have significant effects on corrosion resistance. HR3C, SAVE25 and NF709 are materials for making fire tubes of ultra-supercritical fossil fired plants. They have excellent mechanical performance at temperatures up to 650°C, and show very low corrosion rate even in 650°C SCW (Figure 2d), which satisfy the requirement of SCWR cladding. Surface morphologies of these steels after exposure for 1000 hours show negligible oxide formation, as shown in Figure 4a to c. AL-6XN is a Mo containing austenitic stainless steel. Although Cr content of AL-6XN is only 2% lower than NF709 and SAVE25, it weight gain rate is 2 times higher and an oxide film formed on sample surface (Figure 4d).

Table 1 Chemical compositions of test materials (wt%)

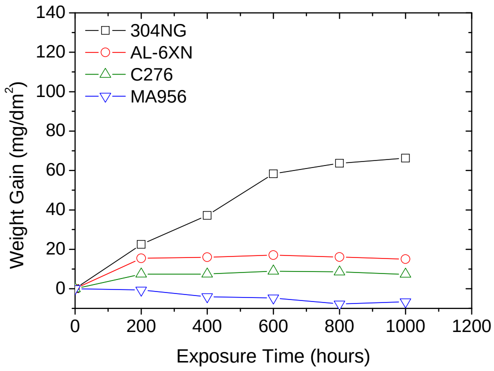
Material	C	Fe	Cr	Ni	Mo	Mn	Si	S	P	Co	W	V	Cu	Al	N	Nb	others
C276	0.001	5.35	15.88	Bal.	15.64	0.52	0.03	0.002	0.005	1.51	3.38	0.02	—	—	—		
F92	0.13	Bal.	9.0	≤ 0.40	0.30 ~0.60	0.30 ~0.60	≤0.50	≤0.010	≤0.020	—	1.50 ~2.00	0.20	≤0.25	≤0.040	0.050	0.07	
AL-6XN	0.020	Bal.	20.43	23.82	6.23	0.42	0.34	0.005	0.024	0.24			0.26		0.211		
304NG	0.018	Bal.	19.4	9.35	—		0.58	0.007	0.018	0.028			0.062		0.089		
MA956	<0.1	Bal.	20.0	—	—	≤0.30			≤0.02	≤0.3	Ti: 0.2/0.6		≤0.15	4.5			Y_2O_3: 0.34
NF709	0.04	Bal.	22	25	1.5	1	0.4	≤0.005	≤0.015	-	-	-	≤0.15	-	0.17	0.25	
HR3C	0.07	Bal.	25	21	0.1	1.1	0.4	≤0.005	≤0.02	-	-	0.07	0.1	-	0.25	0.45	
SAVE25	0.07	Bal.	22.5	18.5	0.1	0.5	0.2	≤0.005	≤0.02	-	1.5	0.04	3.25	-	0.2	0.45	
X20Cr-1	0.1964	Bal.	10.2	0.66	1.01	0.54	0.31	0.0016	0.0092	-	-	0.29	0.028	0.011	-	-	
X20Cr-2	0.1951	Bal.	11.2	0.64	0.99	0.53	0.32	0.0015	0.0092	-	-	0.28	0.029	0.018	-	-	
X20Cr-3	0.1986	Bal.	12.3	0.6419	1.034	0.53	0.30	0.0021	0.0091	-	-	0.31	0.027	0.009	-	-	
B12Cr-1	0.090	Bal.	11.40	0.07	0.46	0.25	0.12	0.003	0.014	2.01	2.10	0.20	0.47	0.011	0.07	0.06	Re:0.06



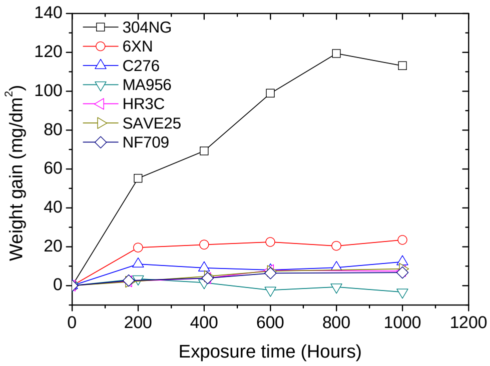
(a) 550℃, in static autoclave



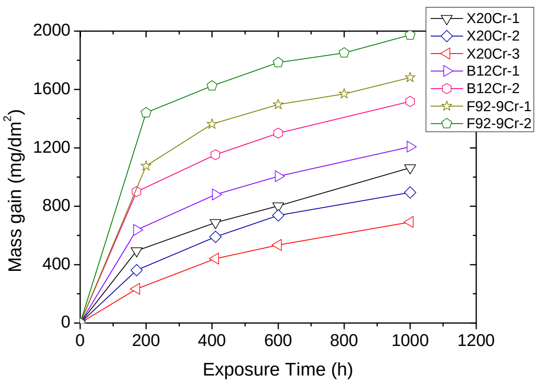
(b) 600℃, in static autoclave



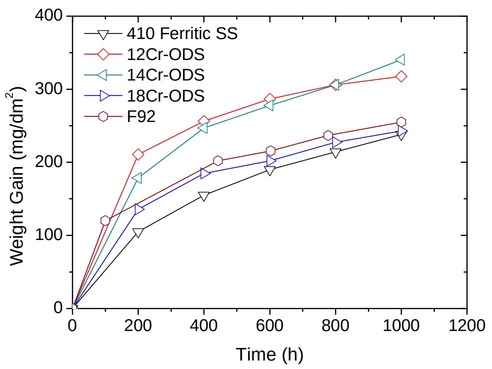
(c) 650℃, in static autoclave



(d) 650℃, in recirculating autoclave



(e) 650℃, F/M steels in circulating autoclave



(f) 550℃, Fe-Cr in recirculating autoclave

Figure 2 General corrosion rates of candidate materials in SCW at 25MPa

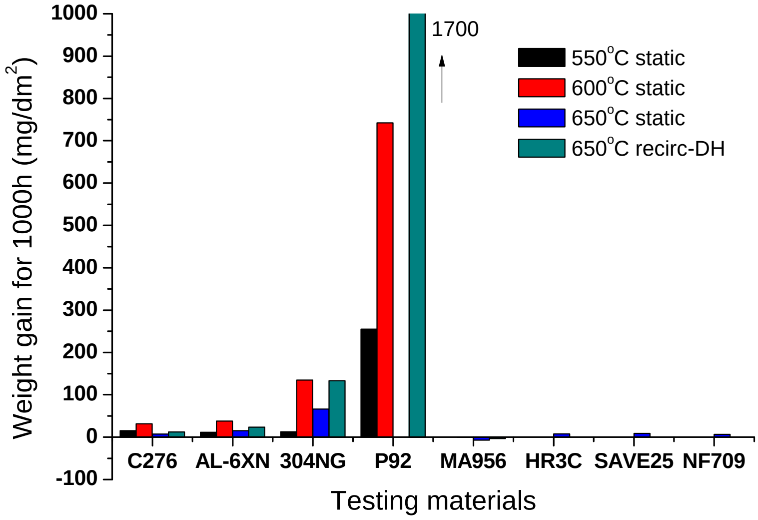
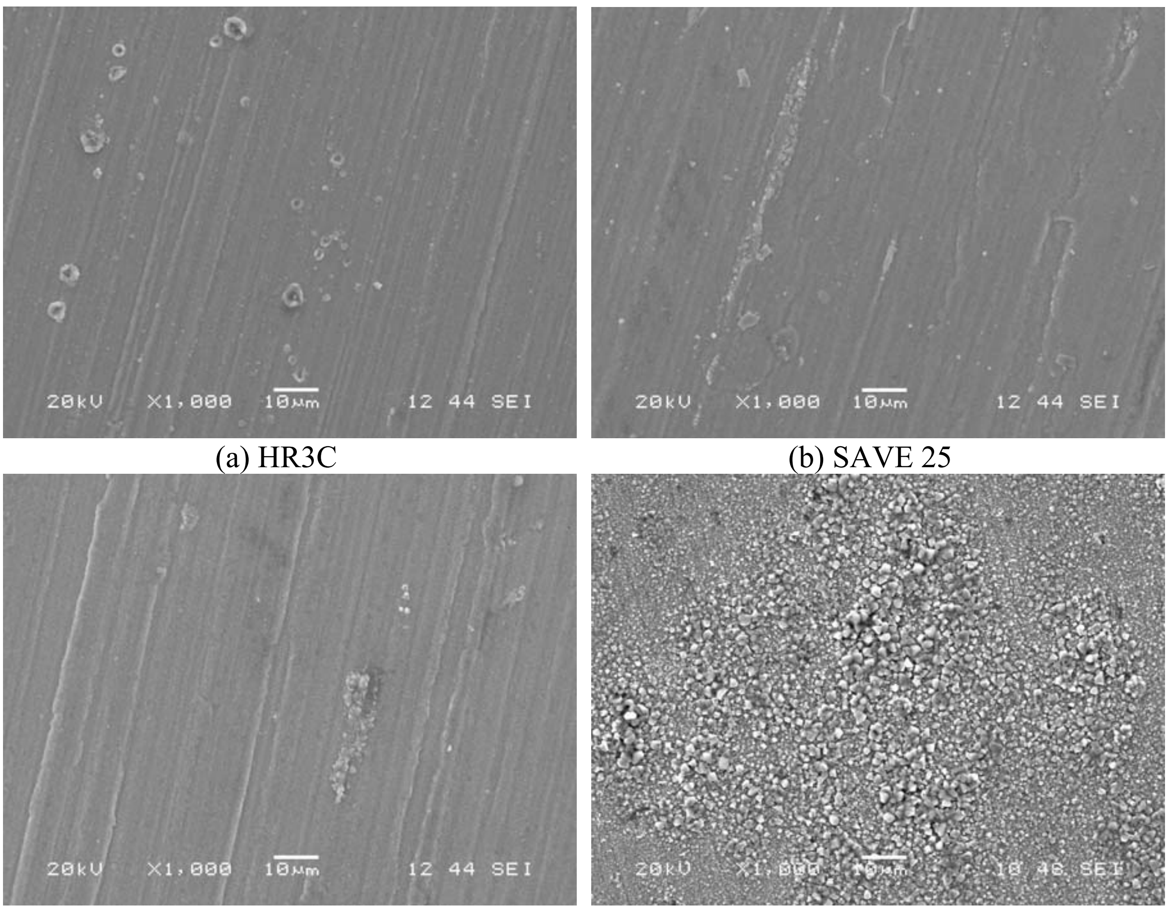


Figure 3 Comparison of general corrosion rate of candidate materials in SCW.



(a) HR3C (b) SAVE 25

(c) NF709 (d) AL-6XN

Figure 4 Surface morphologies of austenitic stainless steels exposed in 650°C, 25MPa supercritical water for 1000 hours

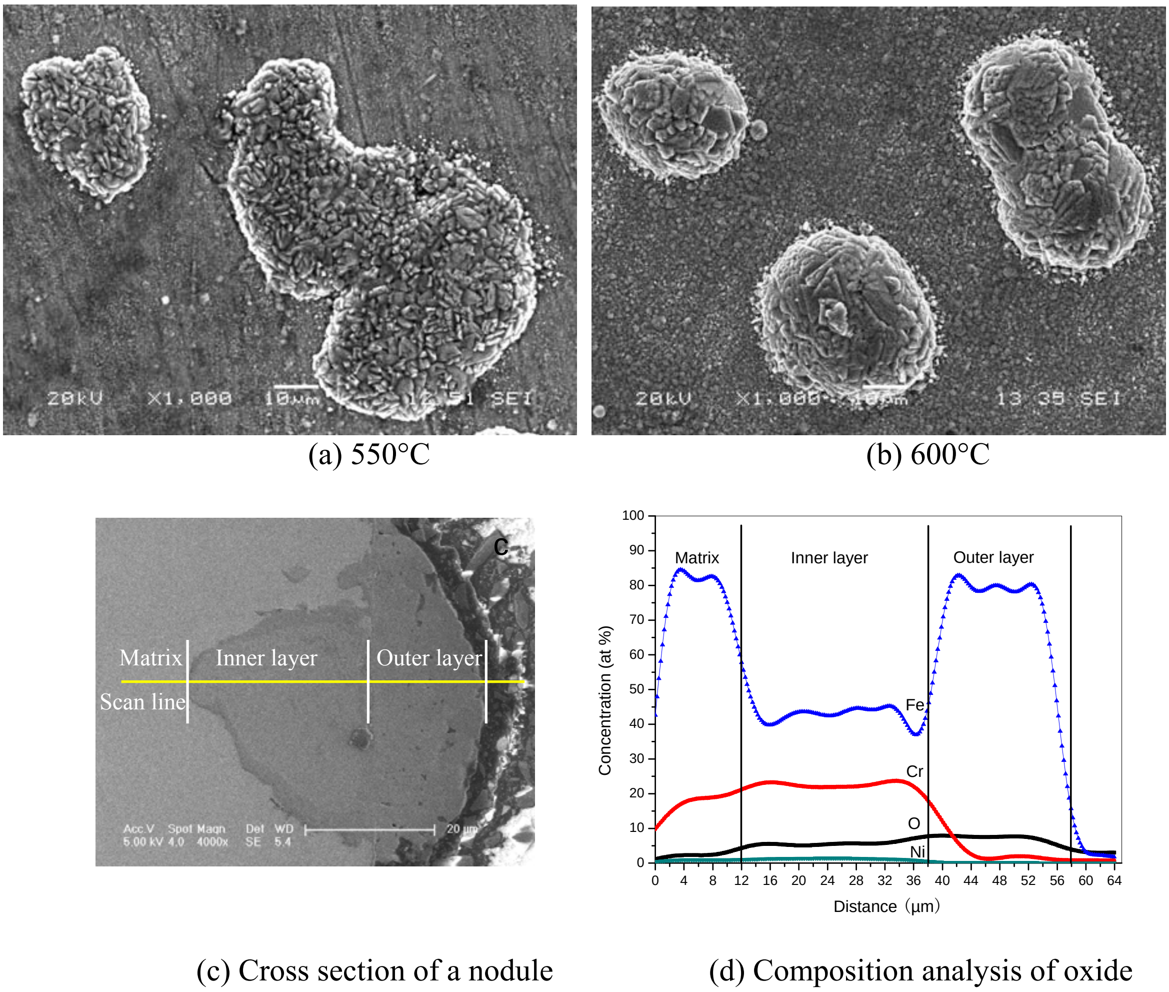


Figure 5 Nodular corrosion of 304NG austenitic stainless steels exposed in supercritical water for 1000 hours

Stainless steel type 304NG is commonly used to make core internals for PWRs. Corrosion test of 304NG in SCW at 550°C showed acceptable performance. However at temperatures above 600°C, the corrosion weight gain rate increases remarkably. A kind of nodular corrosion morphology was observed on the surface, see Figure 5. At temperature of 650°C, exfoliation of some of the nodules can be observed.

The mechanism of formation of nodular corrosion on 304NG is explained clearly in reference [16]. Cr is an element that stabilizes the surface oxide film, preventing oxygen from penetrating into the base metal and preventing Fe from diffusing out to the metal surface. However, Cr can form a volatile compound in high temperature water environments and deplete Cr in the surface oxide film. Diffusion of Cr atoms from the base metal to the surface can make up its loss. The diffusion rate of Cr atoms along grain boundaries is more than 2 orders faster than that through the gain interior. The oxide film above a grain boundary can receive enough Cr atoms by diffusion from the base metal to make up the deficit due to vaporization, while the supply is less than the loss of Cr from the oxide film above the grain interior. In this way, the oxide film above the grain interior is depleted of Cr, and loses its protectiveness. As a result, an oxide island and a corrosion pit grow from the grain interior. Long time exposure in SCW makes the nodules connect and forms a double layered oxide film which have a magnetite top outer layer and a Cr rich spinel inner layer, as shown in Figure 5c and d.

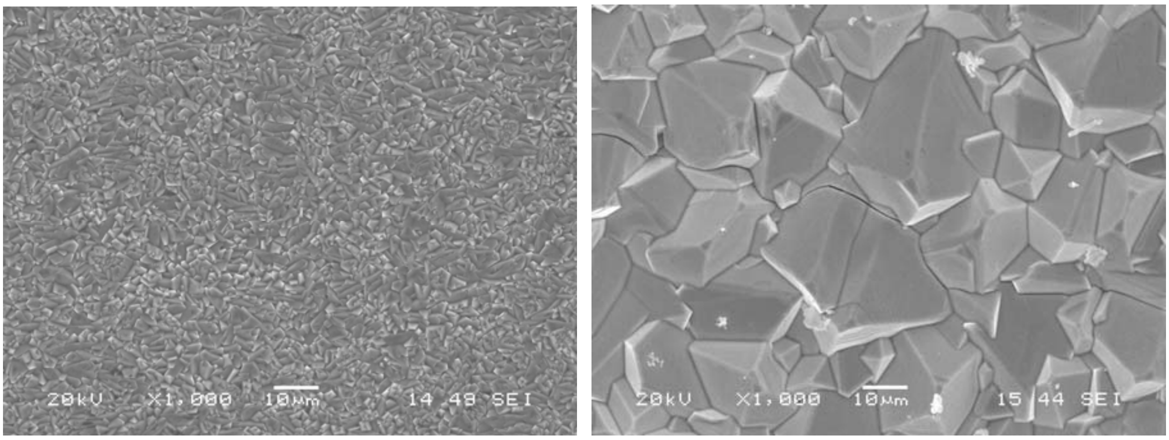
304NG has relatively lower strength at temperature up to 600°C compared with AL-6XN, HR3C, SAVE25 and NF709. Loss of strength and high corrosion rate at high temperatures prevents 304 from being a candidate material for the fuel cladding. However, it can be used to fabricate core internals which operates at low temperatures.

Low swelling austenitic stainless steels originally developed for making FBR fuel claddings such as 316Ti, D9 and PNC1520 were also tested in SCW by other researchers [10],[11],[14]. These materials exhibit high corrosion rates similar to 304NG due to their low Cr concentration, and therefore, the possibility of their application for SCWR fuel cladding is low.

4.3 Corrosion Behaviour of Ferritic/Martensitic Steels

Ferritic/martensitic steels with Cr content varying from 9 to 18 were tested in SCW at temperature from 550 to 650°C, but their performance is not satisfactory for fuel cladding. All of bcc structure ferritic or martensitic steels exhibit high weigh gain rate and form thick oxide films on the surface. The corrosion rate decreases with the increase of Cr concentration in the material, as shown in Figure 2e. The same trend is reported in reference [15].

Crack free oxide film can be formed on ferritic/martensitic steels when exposed in SCW at temperatures below 550°C. However, cracks are observed on oxide films generated in SCW at temperatures above 600°C, as shown in Figure 6. Mismatch in the thermal expansion coefficient between magnetite and base metal cause the cracking of oxide film at higher temperature. It can also be seen that the grain size of oxide film formed at 650°C is rather large, and exfoliation easily occurs.

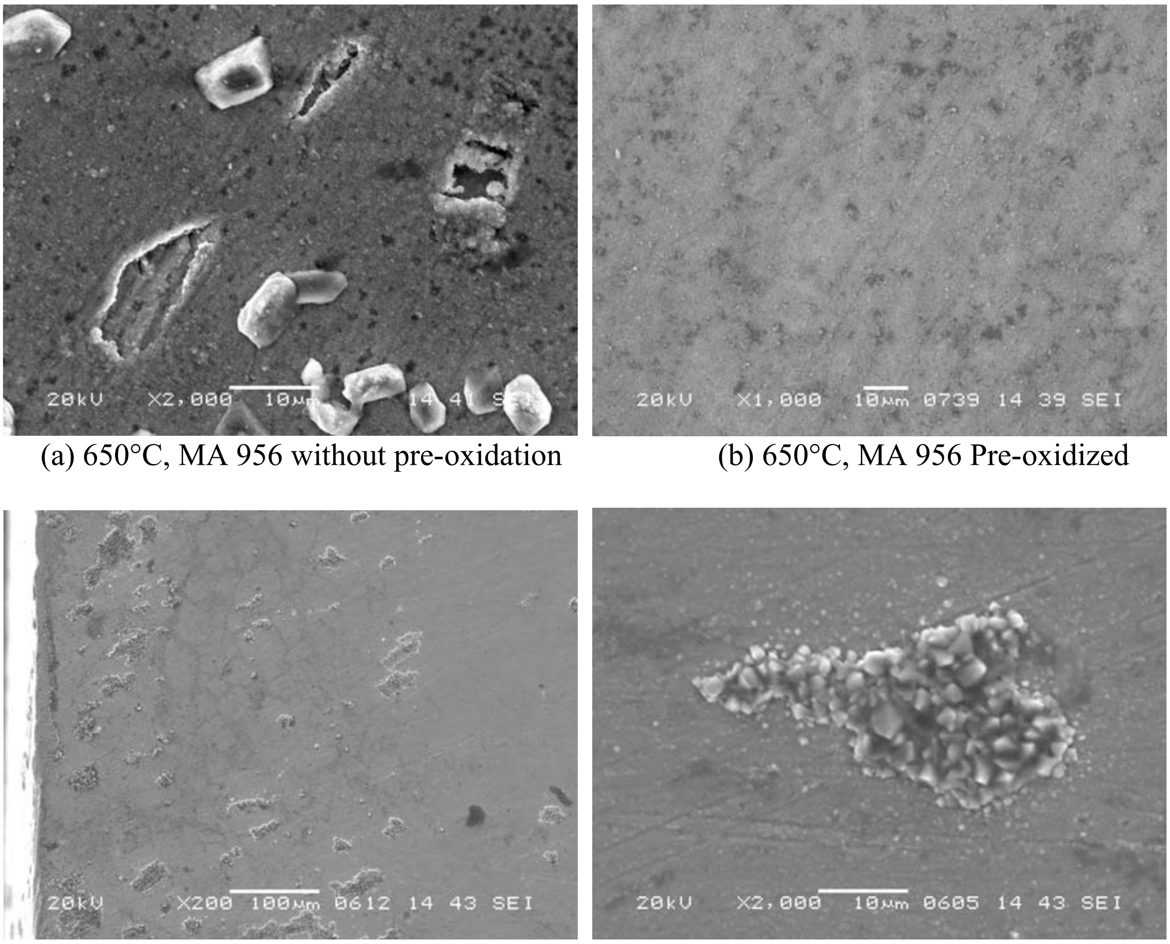


(a) Crack free oxide film formed at 550°C (b) Cracked oxide film formed at 650°C

Figure 6 Oxide film on F/M steel F92 formed in SCW after exposure for 1000 hours

4.4 Corrosion Behaviour of Oxide Dispersion Strengthened (ODS) Steels

Oxide dispersion strengthened (ODS) ferritic/martensitic steels have been developed for application to fuel cladding material for fast breeder reactors (FBR) [17],[18]. ODS steels showed high creep strength at high temperatures due to the dispersion hardening of the oxide particles. ODS steels are also highly resistant to irradiation embrittlement at temperatures between 573 and 773 K up to 15 dpa. However, our corrosion test results show that oxide strengthening does not show beneficial effect in improving the corrosion resistance of materials, as shown in Figure 2f. The weight gain rate of Fe-Cr ODS bcc steels does not follow the Cr relation as shown in Figure 2e. If the ODS steel is not well processed, addition of oxide accelerates the oxidation rate even when Cr content is up to 18%.



(a) 650°C, MA 956 without pre-oxidation (b) 650°C, MA 956 Pre-oxidized

(c) 550°C, Nodular corrosion morphology (d) 550°C, Close view of oxide island

Figure 7 Corrosion of MA 956 after exposure in circulating SCW for 1000h

MA 956 is an alumina-forming high Cr content ODS steel. It shows the lowest general corrosion rate of the materials tested. The nominal chemical composition of MA 965 is Fe-20Cr-4.5Al-0.5Ti-0.5Y_2O_3-0.02C. The presence of Al in the metal forms and maintains a corundum Al_2O_3 scale which is very compact and stable, protecting the matrix from oxidation. Therefore, MA 956 can serve in oxidizing environments at temperatures up to 1100°C. Low corrosion rate and high temperature strength brings it into the priority list of materials for SCWR fuel cladding, although the phase stability should be observed carefully for long time exposure in SCW.

Pre-oxidation is generally required to form the protective surface scale before service. The surface morphologies shown in Figure 7a and b reveals that the pre-oxidized sample maintains its protective film, while the sample without pre-oxidation develop craters on the surface, showing that MA 956 is susceptible to corrosion if the protective film breaks down.

MA 956 shows a weight loss in contrast to the other tested materials, as shown in Figure 2 and Figure 3. Vaporization of the Cr and Al is probably the major reason, especially when pre-oxidation is not performed. Nodular oxide islands are also observed on samples exposed in flowing SCW at 550°C, as shown in Figure 7c and d. The nodular oxide island is Fe_3O_4 (magnetite), and its formation is attributed to the breakdown of the protective alumina in flowing SCW.

4.5 Future Works

In the future corrosion screening tests, candidate materials for fuel cladding will be focus on high Cr concentration alloys such as austenitic stainless steels HR3C, 310, Incoloy 800H, low swelling 316Ti, nickel base alloys such as Alloy 718 and 825, and high-Cr ODS steels such as 18Cr-ODS. Long term general exposure tests will be conducted at temperatures between 500 and 650°C. Stress corrosion cracking, or more precisely, creep induced corrosion cracking, will be studied by using slow strain rate tests in SCW at temperatures from 500 to 650°C. Oxide film stability tests will be conducted in a dynamic circulating loop up to temperature of 650°C to investigate the effects of velocity (up to 25m/s) of SCW fluid on corrosion rate.

5. Conclusion

The major technical requirements of materials for SCWR fuel cladding have been determined based on the estimation according to the current SCWR concepts. Literature review and corrosion tests showed that the most probably fuel cladding material may have an austenitic structure and contain high Cr, concentrations up to 22% or higher, such as HR3C, Aluminium is also helpful to improve corrosion resistance in SCW. The excellent performance in corrosion test shows that alumina-forming high Cr concentration ODS steel, such as MA956, may also be promising for SCWR fuel cladding material.

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