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Review

Corrosion aspects of Ni–Cr–Fe based and Ni–Cu based steam generator tube materials

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A B S T R A C T

This paper reviews corrosion related issues of Ni–Cr–Fe based (in a general sense) and Ni–Cu based steam generator tube materials for nuclear power plants those have been dealt with for last more than four decades along with some updated information on corrosion research. The materials include austenitic stainless steels (SSs), Alloy 600, Monel 400, Alloy 800 and Alloy 690. Compatibility related issues of these alloys are briefly discussed along with the alloy chemistry and microstructure. For austenitic SSs, stress corrosion cracking (SCC) behaviour in high temperature aqueous environments is discussed. For Alloy 600, intergranular cracking in high temperature water including hydrogen-induced intergranular cracking is highlighted along with the interactions of material in various environments. In case of Monel 400, intergranular corrosion and pitting corrosion at ambient temperature and SCC behaviour at elevated temperature are briefly described. For Alloy 800, the discussion covers SCC behaviour, surface characterization and microstructural aspects of pitting, whereas hydrogen-related issues are also highlighted for Alloy 690.

Contents

1. Introduction

Extensive research has been carried out over more than four decades on corrosion related issues of Ni–Cr–Fe based (in a general sense) steam generator (SG) tube materials for nuclear power plants (NPPs). The investigation on Ni–Cu based SG tube material has been limited as compared to Ni–Cr–Fe based alloy. In case of Ni–Cr–Fe based alloys, those considered as SG tube materials for pressurized water reactors (PWRs) systems are austenitic stainless steels (SSs), Alloy 600, Alloy 800 and Alloy 690 [1–15]. For Ni–Cu based alloy, Monel 400 was used as SG tubing in early CANDU reactors and Indian pressurized heavy water reactors (PHWRs) systems [13–15]. Initial SG tube materials for PWRs systems had been austenitic SSs but chloride-induced stress corrosion cracking (SCC) was of major concern that led to discard of this alloy as SG tubing [1,2,4–7,9]. The shortcoming of austenitic SSs was overcome by its replacement, Ni-based Alloy 600, in majority in the 1960s. Incidentally, Coriou and his co-workers confirmed intergranular cracking of Alloy 600 in high temperature pure water in late 1960s while starting preliminary studies in early 1960s [1,10,11]. Intergranular cracking of Alloy 600 primarily led to extensive research on development of an alternative alloy in one side and finding out a remedial heat-treatment of cracking of Alloy

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600 on the other side [5,13–15]. Another alloy that is Alloy 800 containing much lower amount of nickel with much higher amount of iron and slightly more chromium as compared to Alloy 600 was chosen as SG tubing for Kraft Werk Union (KWU) type PWRs and new Indian PHWRs systems due to its good SCC resistance in pure water and chloride solutions [4–7,13,15,16]. Investigation on Alloy 600 has been more extensive as compared to Alloy 800 mainly because of more use of Alloy 600 as well as its more corrosion problems in service [16]. As a result of continued research, Alloy 690 has been developed as a modified version of Alloy 600. Alloy 690 contains lower amount of nickel with higher amount of chromium and possesses better SCC resistance in pure water than Alloy 600 [5,7,8,17,18].

Smooth operation of NPPs depends to a large extent on the trouble-free operation of SGs, which are basically large heat exchangers. To minimize tube failure in SGs, it is of prime importance to understand the compatibility of SG tube materials with the environments in a comprehensive manner. The development of mainly Ni–Cr–Fe based and Ni–Cu based SG tube materials has been driven by the failure of materials owing to their corrosion related issues. Since many new commercial nuclear reactor system designs are emerging, material-environment compatibility related issues particularly in regard to SG systems must be comprehended so that the operation of NPPs remains uninterrupted. To avoid corrosion related problems of nuclear SGs from the past experience, first of all, it is necessary to properly document the trouble-shooting events sequentially. The knowledge gained from the documentation that is preferred in the form of a review report will be useful in avoiding the premature failures of the components. Moreover, the updated review will also elucidate the trend of research. Keeping this in view, an attempt has been made to write a review on corrosion related issues of mainly Ni–Cr–Fe based and Ni–Cu based SG tube materials along with some updated information on corrosion research. This review mainly highlights alloy chemistry, microstructure and material-environment interaction of a few steam generator tube materials. The following sub- sections give material-wise descriptions.

1.1. Austenitic stainless steels

Austenitic stainless steels (SSs) such as Types 304, 316 and similar varieties were initially considered as SG tube materials for PWRs systems. Table 1 gives typical chemical composition of Type 304 L SS. These materials experienced transgranular stress corrosion cracking (TGSCC) in high temperature water containing chlorides [1]. This has led to discarding of austenitic SSs as SG tube materials [2,4–7,9]. SCC behaviour of austenitic SSs has also been studied in high temperature water without chlorides in later period by some researchers. De and Ghosal [6] have investigated the SCC behaviour of Types 304 and 304 L SS in water at 588 K with or without 0.6 ppm Pb and 0.05 or 8 ppm dissolved oxygen (DO). These studies [6] have been conducted to evaluate the effects of impurities like Pb and oxygen on the SCC behaviour of the alloys. Their [6] studies have revealed that Type 304 L SS, irrespective of material condition, is resistant to SCC in high temperature water containing up to 8 ppm DO and 0.6 ppm dissolved Pb, whereas 25% cold worked Type 304 SS suffered TGSCC in the presence of 8 ppm DO without any Pb. The presence of 0.6 ppm Pb in the water has, however, been reported to induce cracking in cold worked Type 304 SS even at very low (0.05 ppm) DO level. Shallow transgranular cracks were noticed in mill-annealed samples of the SS in water containing 8 ppm DO and cracking was prevented by stress relieving treatment [6]. Dutta et al. [19] have studied the effect of heat-treatments on SCC behaviour of nitrogen-containing austenitic 316 L SSs in high temperature water. It was indicated from constant-strain tests of these two SSs (one containing 0.16%N and the other 0.19%N) in 533 K water containing chlorides that the cracking mode (intergranular or transgranular) is microstructure-dependent. An ageing treatment at 1023 K for 7.5 h (27 Ks) that results in sensitization makes these two nitrogen-containing SSs susceptible to intergranular stress corrosion cracking (IGSCC) as revealed by slow strain rate tensile (SSRT) tests in 523 K water [19].

Table 1
Typical chemical composition of Type 304 L SS (wt%).

C	Cr	Fe	Ni	Mn	Si
0.03	18	Bal	10	0.6	0.6

Table 2
Typical chemical composition of Alloy 600 (wt%).

C	Cr	Fe	Ni	Mn	Si	Cu	S	P
0.03	15.1	7.6	Bal	0.35	0.21	0.01	0.007	0.009

1.2. Alloy 600

To overcome the deficiencies (chloride-induced SCC) of austenitic SSs, high-nickel alloys were used as replacements. In most cases, Alloy 600 found application as a SG tube material for PWRs in the 1960s [1,10]. Alloy 600 is a solid solution-strengthened, austenitic alloy containing about 75% Ni, 15% Cr and 7% Fe as major alloying elements. Table 2 shows typical chemical composition of Alloy 600.

In late 1960s, SCC of Alloy 600 from the primary side was detected in high purity water. This was termed as Coriou cracking after the name of the researchers Coriou and his co-workers [1,10,11] and now popularly known as primary water stress corrosion cracking (PWSCC). It is to be mentioned that Coriou et al. indicated the phenomenon in their preliminary studies in early 1960s but confirmed in late 1960s [1,10]. This type of intergranular cracking and SCC from the secondary side due to crevice and denting effects were the main causes of tube failures. Intergranular cracking of Alloy 600 mainly led to extensive research on development of a better alloy in one side and finding out a remedial heat-treatment to cracking of Alloy 600 on the other side [5]. Later on, a thermal treatment (TT) of Alloy 600 at around 973 K for 10–15 h (36–54 Ks) designated as 600TT and developed jointly by Electricité de France (EdF), Framatome and Westinghouse Electric Corporation (WEC) was found to improve the SCC resistance of the alloy [5].

Alloy 600 has been studied quite extensively. Investigations in the later period [20–25] on Alloy 600 relating to hydrogen have indicated that this alloy is susceptible to hydrogen-induced intergranular cracking. NiO film formation on the surface of the alloy is responsible for the occurrence of hydrogen-induced intergranular cracking [22]. During NiO film formation, nascent hydrogen is absorbed by the alloy. Hydrogen-induced intergranular cracking of Alloy 600 is induced by the nucleation and growth of a fine array of grain boundary microvoids in front of an advancing crack [23]. The results of Sui et al. [26] indicate that Alloy 600, subjected to low-temperature mill-annealing treatment, exhibits intergranular cracking in a hydrogen/steam environment at 653 K.

Besides hydrogen-induced intergranular cracking, environmentally-assisted cracking of Alloy 600 has also been reported by several researchers later on. De and Ghosal [6] have studied SCC behaviour of Alloy 600 in water at 588 K, with or without 0.6 ppm Pb and 0.05 or 8 ppm DO. The authors [6] have noticed intergranular cracks in highly stressed Alloy 600 after an exposure of 1600 h (5.76 Ms), irrespective of Pb and DO content in the water.

It is indicated that Alloy 600, heat-treated at 873 K for 24 h (86.4 Ks) or longer time after annealing treatment, is resistant to SCC. This could be attributed to semi continuous intergranular precipitation caused by the heat-treatment at 873 K [6]. Was and Lian [27] have examined the roles of carbides in SCC resistance of Alloy 600 and controlled purity Ni–16%Cr–9%Fe in primary water at 633 K. A grain boundary carbide microstructure has been found to be more resistant to IGSCC as compared to an intragranular carbide microstructure and crack growth rate has been found to increase with increasing strain rate [27]. Angeliu et al. [28] have investigated repassivation and crack propagation of Alloy 600. The authors [28] have observed a reduction in crack growth of Alloy 600 in 561 K water at a constant stress intensity factor of 27.5 MPa $m^{1/2}$ as the level of dissolved hydrogen increased from zero to 18×10^{-6} m^3/kg (0–1 ppm). Slip-oxidation is the mechanism for crack advancement in which mechanism, crack advances through an iterative process of surface film rupture during straining that exposes the material to corrosive environment and passivation by film formation [28]. The effect of cyclic loading on environmentally-assisted cracking of Alloy 600 in typical nuclear coolant waters has been reviewed by Congleton et al. [29]. The review [29] shows that Alloy 600 exhibits enhanced crack growth rates relative to those in air when tested in PWR primary coolant and in oxygenated pure water. The enhancements are found to be larger in oxygenated pure water as compared to that in PWR primary coolant [29]. Kamaya and Totsuka [30] have investigated stress corrosion crack propagation of Alloy 600 in simulated PWR primary coolant and have concluded that interaction between multiple cracks might be a key factor in crack growth acceleration after 1500 h (5.4 Ms). The inhibitory effect of boric acid on intergranular attack (IGA) and SCC of Alloy 600 in high temperature water has been examined by Kawamura et al. [31]. The addition of boric acid has been found to have a slight retarding effect on crack velocity in both all-volatile treatment water and caustic environments [31].

McIntyre et al. [32] have examined the effects of pH excursions on the surface film composition of Alloy 600. The authors [32] have noticed that exposure of the alloy to acidic sulfate (simulating abnormal secondary side SG coolant condition) results in film formation that is less protective as compared to that formed under normal SG condition. A return to an alkaline condition (near-normal SG condition) after an acid excursion has been reported to result in some sulfide deposition on Alloy 600 surface [32]. Due to sulfide deposition, the composition of surface film and its stability are likely to be affected and this will influence corrosion resistance of the alloy.

1.3. Monel 400

Monel 400 is a Ni–Cu based single-phase alloy. Typical chemical composition of Monel 400 is shown in Table 3. Monel 400 exhibits excellent resistance to many corrosive environments. This alloy, unlike austenitic SSs, has been found to be resistant to chloride-induced SCC [33]. Monel 400, irrespective of material condition, has also been reported to be resistant to SCC in high temperature water containing up to 8 ppm DO and 0.6 ppm dissolved Pb [6]. Monel 400 has been extensively used as a heat exchanger tube material. This alloy was employed as a SG tube material in early CANDU reactors and early Indian PHWRs systems due to its good corrosion resistance in low oxygen environments [13–15]. However, this alloy has been reported to be susceptible to intergranular corrosion (IGC) associated with pitting under certain conditions [33]. The effect of heat-treatments on corrosion behaviour of Monel 400 in acidic sulfate solutions at ambient temperature with different pH levels and Cl^- contents has been investigated by De and Bose [33]. It is indicated from their study [33] that the specimens heat-treated at 1173 K for 1 h (3.6 Ks) followed by air cooling, displayed passive behaviour in 0.5 M Na_2SO_4, whereas addition of 0.2% $CuCl_2$ to this solution of pH 3 enhances IGC and pitting attack. The samples, heat-treated at 1173 K for 1 h (3.6 Ks) followed by water quenching, have shown intense IGC on exposure to 0.25 M H_2SO_4 + 0.2% $CuCl_2$ solution of pH 3. On the other hand, furnace-cooled specimens have been reported to exhibit the least IGC and a heat-treatment at 973 K for 65 h (234 Ks) has indicated reduction in IGC susceptibility of the alloy in 0.25 M H_2SO_4 + 0.2% $CuCl_2$ solution of pH 3. Type of ageing treatment and cooling rate are the key factors in regulating the grain boundary structure of the alloy in terms of P and S distribution [33]. The addition of a small amount of Hf (0.4%) as an alloying element to this alloy has been found to have a beneficial effect on the IGC resistance of the alloy that could be attributed to combination of Hf with S reducing the segregation of latter at the grain boundaries [33].

The effects of pH excursions on the surface film composition of Monel 400 have been examined by McIntyre et al. [32]. The behaviour of the alloy is found to be similar to that of Alloy 600 as discussed earlier.

It is to be mentioned that Monel 400 is still in use as SG tubing in some reactor systems (in old Indian PHWRs and some other systems). In new designs, this alloy has been replaced by Alloy 800 that will be discussed below.

Table 3
Typical chemical composition of Monel 400 (wt%).

Ni	Cu	C	Fe	Mn	Si	S	P
68.5	25.2	0.09	1.2	0.76	0.004	0.007	0.026

1.4. Alloy 800

Chloride-induced transgranular cracking of austenitic SSs led to their substitution with Alloy 600 in most of the cases and with Alloy 800 in some cases [5–7,16]. Alloy 800 is an austenitic, precipitation-hardenable type alloy containing about 45% Fe, 30% Ni and 20% Cr as major alloying elements. Minor alloying elements like Ti and Al are added to this alloy to bring about precipitate Ni_3(Ti,Al), known as gamma prime, on longer ageing to induce precipitation hardening effect. Table 4 shows typical chemical composition of Alloy 800. Fig. 1 shows grain boundary Cr-carbide precipitates in Alloy 800 caused by ageing the alloy at 973 K for 10 min (0.6 Ks) followed by air cooling. Fig. 2 shows large, faceted primary TiN (hexagonal structure) particles randomly distributed in the alloy. These primary nitride particles have been found to act as precipitating sites for secondary Cr-carbides, which are of $M_{23}C_6$ type. Alloy 800 was chosen as a SG tube material for KWU type PWRs and new Indian PHWRs owing to its very good resistance to SCC in pure water and chloride environments [13,15]. Alloy 800, irrespective of material condition, has also been reported to be resistant to SCC in high temperature water containing up to 8 ppm oxygen and 0.6 ppm dissolved Pb [6].

The surface film, which is formed on austenitic Alloy 800 at the onset of passivity in an acidic chloride environment at ambient temperature, has been characterized [34] using X-ray photoelectron spectroscopy (XPS). The study has revealed that the surface film consists of Cr^{+3} (as Cr_2O_3) without any Fe^{+3}/Fe^{+2} or Ni^{+2}. In normal and abnormal SG coolant conditions [32], the behaviour of the alloy is reported to be similar to that of Alloy 600 as mentioned earlier. An exposure of cold worked (~50%) Alloy 800 in 673 K steam (initial pH of water was 10.1) for a period of 264 h (0.9504 Ms) indicates almost nil corrosion rate [35]. Scanning electron microscopy (SEM) reveals a number of small oxide particles on the surface exposed in steam indicating initiation of oxide formation. XPS has indicated that this surface contains mixed oxides of

Table 4
Typical chemical composition of Alloy 800 (wt%).

C	Ni	Cr	Fe	Ti	Al	Mn	Si	S	P	Cu	N
0.035	30.1	19.5	Bal	0.5	0.3	0.72	0.4	0.012	0.007	0.07	0.02

chromium and iron along with the elemental form of chromium, iron and nickel. Fig. 3 shows SEM micrograph revealing a number of small, oxide particles and a large, faceted TiN particle on the surface of cold worked Alloy 800 [35] that has been exposed in 673 K steam (initial pH of water was 10.1). The energy dispersive X-ray analyses of the large particle are also shown.

A study [34] relating to microstructural aspects indicates that the large, faceted primary TiN (hexagonal structure) particle/austenite matrix interface in the alloy may provide a preferred site for pit initiation in the chloride and acidic chloride environments at ambient temperature. No preferential attack has been observed at the small, spherical-shaped primary TiC (face centered cubic structure) particles. According to the investigators [34], this could be attributed to their (TiC particles) very little misfit with the austenite matrix due to their smaller size and spherical shape.

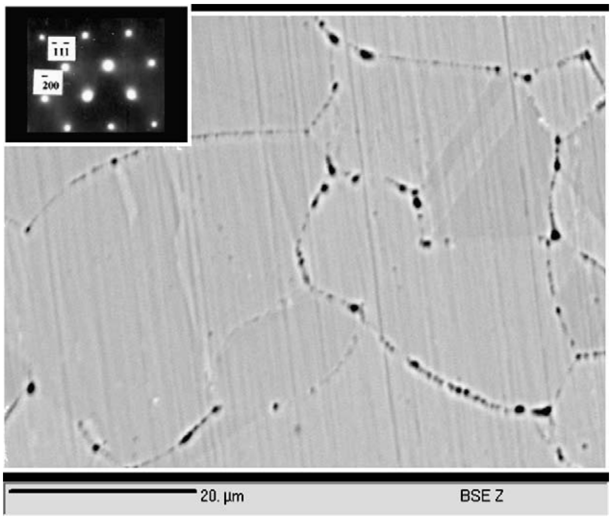


Fig. 1. Intergranular Cr-carbides in aged Alloy 800. Inset shows diffraction pattern of matrix (fcc).

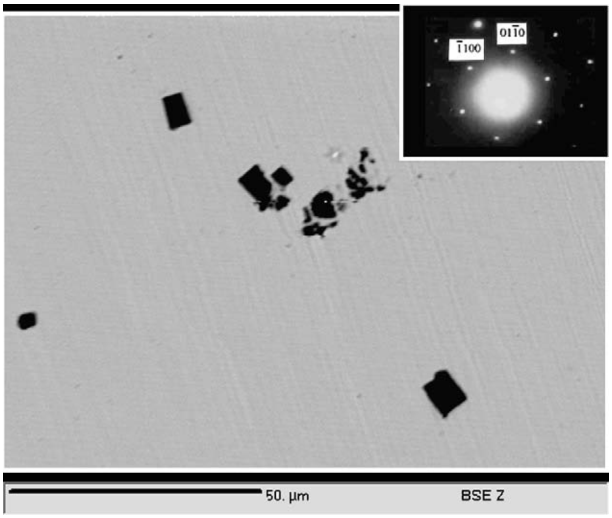


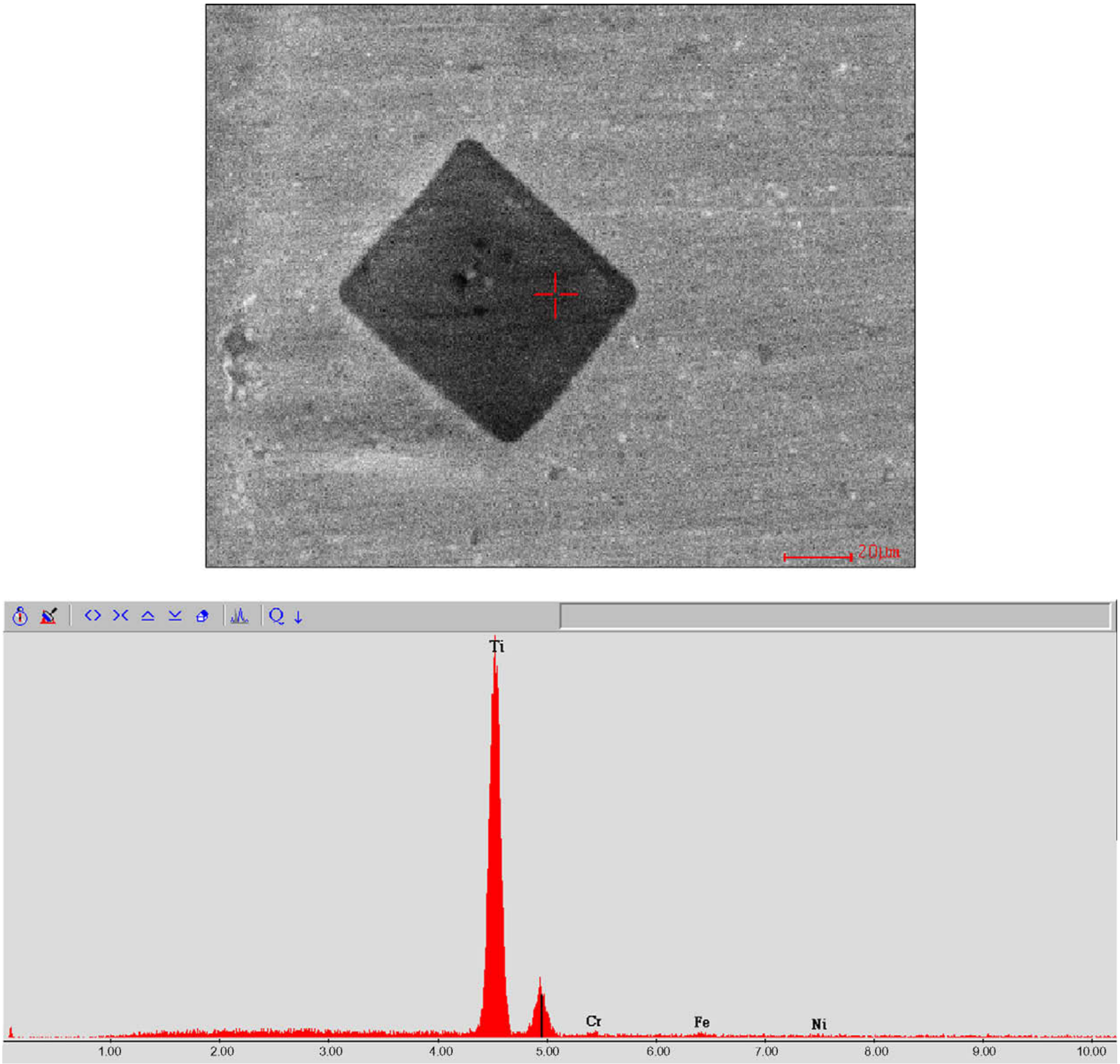
Fig. 2. Primary TiN particles in Alloy 800. Inset shows diffraction pattern of particle (hexagonal).

1.5. Alloy 690

Extensive research has, however, led to the development of another modified alloy that is Alloy 690. This alloy is a solid solution-strengthened, austenitic alloy containing about 60% Ni, 28% Cr and 8% Fe as major alloying elements. Table 5 shows typical chemical composition of Alloy 690. The alloy has been found to contain primary TiC (small, spherical-shaped) and TiN (large, faceted) particles randomly distributed in the austenite matrix [36–38]. Thermal treatment of the alloy at 973 K for 4 h (11.4 Ks) forms discrete grain boundary Cr-carbide precipitation along with intragranular Cr-carbides, whereas ageing treatment for 16 h (57.6 Ks) results in intragranular and near continuous intergranular Cr-carbide precipitation [36,38]. These secondary Cr-carbides are of $M_{23}C_6$ type and large, faceted TiN particle/matrix interface has been reported to act as a precipitation site during ageing of Alloy 690 [36,38]. Fig. 4 shows high-resolution transmission electron micrograph of solution-annealed (1393 K/1.8 Ks, water quenched) specimen revealing austenite matrix, large, faceted primary TiN particle and particle/matrix interface. {1 1 1} planes of austenite matrix (face centered cubic structure) having interplanar spacing of 0.2 nm are found to be discontinuous at the large TiN particle (hexagonal structure).

Alloy 690 possesses superior SCC resistance in pure water as compared to Alloy 600. However, the alloy has also been reported to be susceptible to SCC and IGA in deaerated caustic environments [17,39]. Thermal treatment of Alloy 690 at around 973 K that brings about semi continuous grain boundary Cr-carbide precipitates along with intragranular Cr-carbides has been reported by several researchers to improve the caustic corrosion resistance of the alloy as discussed at a length by Dutta et al. [36,38]. The relationship between the resistance to IGA and IGSCC and the chromium depletion of Alloy 690 has been investigated by Yu and Yao [40]. Higher equilibrium chromium concentration at the intergranular carbide/matrix interface results in good resistance to IGA and IGSCC [40]. It is also reported [40] that heat-treatment at 811–1073 K for 0.5–100 h (1.8–360 Ks) has no pronounced effect on the resistance to IGA of the alloy in boiling 65% nitric acid solution. Kai et al. [41] have also noticed similar behaviour. High chromium content of the alloy results in its superior resistance to IGA and SCC [41].

Studies relating to hydrogen embrittlement of Alloy 690 have received some attention. The samples subjected to low-temperature mill-annealing treatment, exhibit intergranular cracking in a hydrogen/steam environment at 653 K [26]. Younes et al. [42] have noticed that the heats of Alloy 690 are susceptible to hydrogen embrittlement to a lesser extent than those of Alloy 600. Brown and Mills [43] have observed that the decrease in fracture resistance of Alloy 690 in 327 K water is associated with a fracture-mechanism transition from dimple rupture to intergranular cracking. Hydrogen generated by a corrosion reaction within the crack is responsible for the low-temperature embrittlement in water [43]. Present author with his co-workers [37] have investigated the susceptibility of Alloy 690 to hydrogen embrittlement and have noticed an indication toward hydrogen-induced ductility loss for the samples that is believed to be due to a



EDX Analyses		
Large, faceted undissolved particle		
Element	Wt%	At%
Ti	95.99	96.51
Cr	1.23	1.14
Fe	1.58	1.37
Ni	1.20	0.99

Fig. 3. SEM micrograph and EDX analyses of the surface of cold worked Alloy 800 exposed in 673 K steam (initial pH of water was 10.1). A number of small oxide particles (whitish in colour) and a large, faceted particle are noticed. The EDX analyses of the large particle are also shown.

Table 5
Typical chemical composition of Alloy 690 (wt%).

C	Cr	Ni	Fe	Ti	Mn	Si	S	P	Cu	N
0.01	27.7	Bal	8.5	0.3	0.18	0.13	0.002	0.007	0.06	0.1

hydrogen-enhanced microvoid growth process. As microvoid growth process occurs at the last stage of fracture, the effect of hydrogen on ductility loss is not substantial [37].

In normal and simulating abnormal secondary side SG coolant conditions [32], the behaviour of the alloy is found to be similar

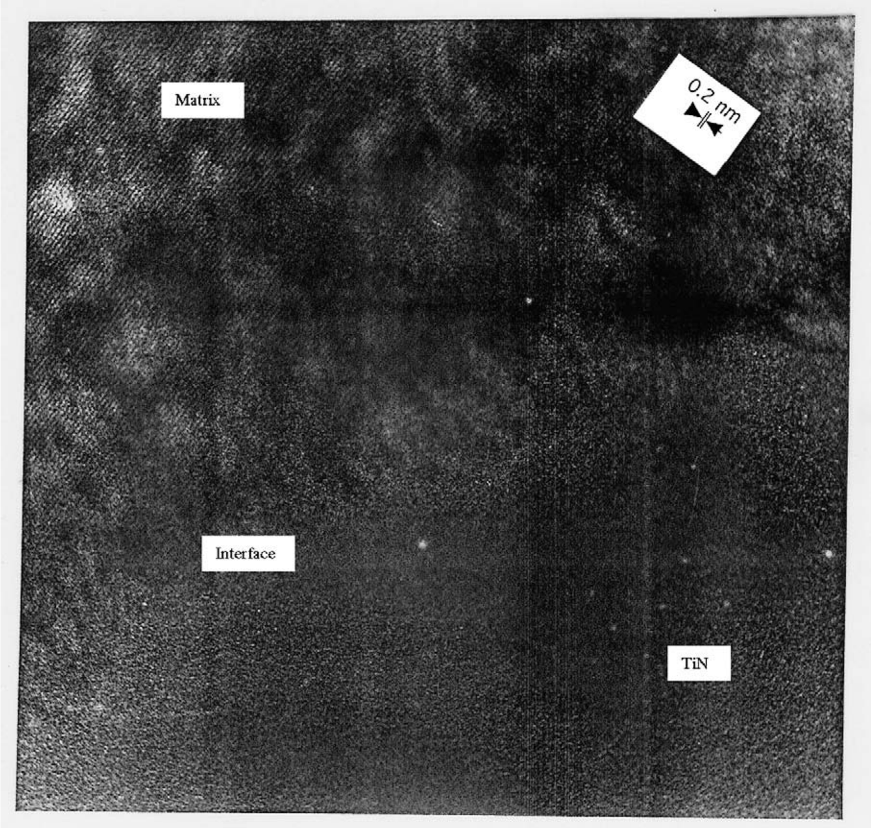


Fig. 4. High-resolution transmission electron micrograph of solution-annealed Alloy 690 revealing austenite matrix, large, faceted primary TiN particle and particle/matrix interface.

to that of Alloy 600 as stated earlier. Surface films those formed on the alloy at the onset and later stage of passive region in an acidic chloride environment at ambient temperature have been characterized [37] using XPS. The studies revealed [37] that the surface film formed at the onset of passivity consists of $Cr(OH)_3$, nickel in a transition state of Ni^{+2} and Ni^0 but without any Fe^{+3}/Fe^{+2}. The passive film formed on the alloy at the higher anodic potential has been found to consist of Cr_2O_3 without any Fe^{+3}/Fe^{+2} or even Ni^{+2}/Ni^0.

Studies [36,37] on microstructural aspects of the corrosion revealed that the large, faceted primary TiN (hexagonal structure) particle/austenite matrix interface may act as a preferential site for pit initiation in the acidic chloride environments at ambient temperature, whereas no preferential attack has been noticed at the small, spherical-shaped primary TiC (face centered cubic structure) particles. This could be related [36,37] to their (TiC particles) very little misfit with the austenite matrix owing to their smaller size and spherical shape, which is not in the case for large TiN particles as is evident from Fig. 4. In caustic environments, similar behaviour of undissolved primary TiC and TiN particles has also been noticed [38].

2. Conclusions

The following conclusions can be drawn from the present review:

(1) In Ni–Cr–Fe systems, development of SG tube materials has taken place mainly because of corrosion that is material-environment compatibility related issues.
(2) Alloy 600 has been studied quite extensively mainly because of its extensive use as SG tubing and experience of corrosion related problems in service.
(3) Studies relating to evaluation of surface film composition of high-nickel SG tube materials have received attention to a lesser extent as compared to other corrosion studies.
(4) Initially, corrosion research on SG tube materials was more or less confined to failure-related problems but later on, the trend has been on understanding the basic mechanisms as well as microstructural aspects.
(5) A number of years have passed since Alloy 690 was developed. Alloy 800 has also been developed long back. It is desirable to develop some better materials for better performances.

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