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Stress Corrosion Cracking Sensitivity of High Purity Fe-Cr Alloys in High Temperature Water

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Abstract: Stress Corrosion Cracking (SCC) sensitivities of high purity Fe-Cr alloys were investigated in simulated Boiling Water Reactor (BWR) and primary Pressurized Water Reactor (PWR) cooling waters, by constant extension rate tests (CERT) at a strain rate of $4 \times 10^{-7} \text{ s}^{-1}$. No high purity Fe-Cr alloy showed any SCC sensitivity in either simulated BWR and PWR primary water environment.

1. INTRODUCTION

Stainless Steels are widely used for structural materials such as piping and equipment in nuclear and fossil fuel power plants. In particular, high corrosion resistance in high-temperature cooling water is required as one of the most important material characteristics.

Generally, stress corrosion cracking (SCC) is considered to occur in cases when the three factors comprising material, stress and environment interact with one another [1,2]. The authors studied the SCC behaviour of materials in simulated nuclear and fossil cooling water environments [3-7]. Dynamic strain enhances SCC sensitivity in corrosive environments due to the mechano-chemical effects [8,9].

The Japan Research Association on Ultra High Purity Materials has promoted collaborative study of high purity Fe-Cr alloys as one of its research activities [10].

In the present work, the SCC sensitivity behaviour of high purity Fe-Cr alloys were investigated in the simulated BWR and PWR primary cooling waters, as compared with those of one intermediate purity Fe-Cr alloy and commercial-base type 430 and 304 stainless steels.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The chemical composition of specimens for tests is shown in Table 1. Four kinds of high purity Fe-Cr alloys with Cr contents of 16, 25, 35 and 50wt% from high purity electrolytic iron and chromium, an intermediate purity Fe-30wt%Cr, and commercial base type 430 and 304 stainless steels were used for the tests.

Material	Cr	Ni	C	S	O	N	Mn	Si	Al	P
Fe-16Cr	16	na	0.0005	0.0001	0.0076	0.0006	na	na	na	na
Fe-25Cr	25	na	0.0006	0.0002	0.0070	0.0008	na	na	na	na
Fe-35Cr	35	na	0.0008	0.0004	0.0081	0.0009	na	na	na	na
Fe-50Cr	50	na	0.0009	0.0005	0.0070	0.0011	na	na	na	na
Fe-30Cr	30	na	0.012	0.0006	0.0084	0.0084	0.10	0.11	0.036	0.028
Type430	16.1	0.1	0.06	0.001	na	na	0.14	0.50	na	0.016
Type304	18.5	9.4	0.07	0.001	na	na	1.50	0.50	na	0.002

Table 1 Chemical composition of specimens (wt.%)

Fig.1 Specimen Size(in mm) and Shape for Constant Extension Rate Test

Fig.2 Flow Diagram of Stress Corrosion Test Facility

2.2 Test Specimens and Heat Treatment

The size and shape of specimens for mechanical and stress corrosion cracking tests are shown in Fig.1.

Solution heat treatment of all the specimens was conducted by heating at 1000°C for 30 min and then cooling in ambient air. After this solution treatment, the specimens were subjected to sensitization heat treatment which is required for the simulated welding heats in power plant components and piping by heating at 650°C for 2hr and then cooling in air.

2.3 SCC Test

The SCC test was conducted in a recirculated-refreshed water loop shown in Fig. 2. For the simulated BWR environment, the high purity water at 288°C was supplied to the test vessels (autoclaves). The quality of water at the outlet of the autoclave is pH6.7, the electrical resistivity 0.6 μ S/cm and the dissolved oxygen (DO) approximately 8ppm, controlled by flow of mixed N₂+O₂ gas. The DO concentration in the usual BWR feedwater is 0.2ppm (200ppb) on average. The 8ppm DO concentration in this work was therefore increased during an environmentally-accelerated SCC test.

For the simulated PWR primary environment, water at 320°C containing about 500ppm B (H₃BO₃), 2ppm Li(LiOH) and 30cc/l H₂ was supplied to the autoclaves. The quality of water at the outlet of the autoclave was pH6.9, the electrical resistivity 21 μ S/cm, 2.5ppm H₂ content and the dissolved oxygen content less than 5ppb. This coolant environment of the present test was not accelerated for corrosion, as compared to the actual PWR primary water.

In the water environments above-mentioned, the constant extension rate test (CERT) / slow strain rate test (SSRT) was conducted at the strain rate of 4x10⁻⁷sec⁻¹ for the SCC evaluation.

2.4 Tensile Test in a Non-corrosive Environment

In order to compare the mechanical properties and fracture mode in water with those in non-corrosive environments, CERT/SSRT was also conducted in Ar gas, purity 99.99vol.%. The tensile testing machine was the same one used in the SCC tests. The strain rate was also the same value of 4x10⁻⁷sec⁻¹ as for the SCC tests.

2.5 Observation after Testing

The fracture mode of the specimens after SCC and tensile tests was observed by SEM.

3. RESULTS AND DISCUSSION

3.1 Mechanical Properties

The results of the tensile tests, which were conducted in the non-corrosive pure Ar gas at 288°C and 320°C, are summarized in Tables 2 and 3, respectively. At first, both 0.2% proof stress and the ultimate tensile strength of high purity Fe-Cr alloys become high with Cr concentration, although those at different test temperatures were almost the same. This behaviour may be due to the solid solution hardening effects of the Cr atom. Total elongation of high purity Fe-Cr alloys shows the behaviour under investigation. The Fe-35Cr alloy has the largest elongation, and Fe-50Cr alloy has astonishingly high ductility.

On the other hand, the elongation of intermediate purity Fe-30Cr alloy is a little lower than that of high purity Fe-35Cr alloy.

Material	0.2%Proof Stress (MPa)	Ultimate Tensile Stress (MPa)	Total Elongation (%)
Fe-16Cr	112	278	27.3
Fe-25Cr	115	364	38.9
Fe-35Cr	180	447	40.5
Fe-50Cr	542	678	18.6
Fe-30Cr	182	448	34.2
Type430	336	510	14.7
Type304	164	521	54.2

Table 2 Constant strain rate test results, at a strain rate of 4x10⁻⁷sec⁻¹ in 288°C pure Ar

Material	0.2%Proof Stress (MPa)	Ultimate Tensile Stress (MPa)	Total Elongation (%)
Fe-16Cr	134	276	29.9
Fe-35Cr	174	436	38.3
Fe-50Cr	519	661	17.5
Type430	219	462	20.0
Type304	182	534	50.5

Table 3 Constant strain rate test results, at a strain rate of $4 \times 10^{-7} \text{sec}^{-1}$ in pure Ar at 320°C

As for the characteristics of the high purity Fe-16Cr alloy and the Type430 ferritic stainless steel which have almost the same Cr content, the former material has higher elongation and lower strength than the latter. This mechanical behaviour of high purity Fe-16Cr alloy mainly results from the low content in interstitial elements such as carbon.

Detailed consideration and interpretation of the above-mentioned results will be clarified from the viewpoint of these microstructures and crystal structure in the near future.

The mechanical properties of commercial base Type430 and 304 stainless steels show the usual behaviour in both strength and ductility.

3.2 SCC Sensitivity

The results of the SCC test in pure water at 288°C (simulated BWR coolant) and water at 320°C (simulated PWR primary coolant) are shown in Tables 4 and 5, respectively. Stress-strain curves tested at 288°C in high purity Ar gas and in the simulated BWR are shown in Fig. 3 and 4, respectively.

Material	0.2%Proof Stress (MPa)	Ultimate Tensile Stress (MPa)	Total Elongation (%)	Stress Corrosion Cracking Sensitivity
Fe-16Cr	116	310	32.7	Non
Fe-25Cr	129	373	36.1	Non
Fe-35Cr	186	440	42.0	Non
Fe-50Cr	506	629	18.6	Non
Fe-30Cr	187	456	31.5	Non
Type430	301	502	15.9	Non
Type304	167	388	17.4	IGSCC *

IGSCC *: Intergranular Stress Corrosion Cracking

Table 4 Constant strain rate test results, at a strain rate of $4 \times 10^{-7} \text{sec}^{-1}$ in pure water at 288°C

Material	0.2%Proof Stress (MPa)	Ultimate Tensile Stress (MPa)	Total Elongation (%)	Stress Corrosion Cracking Sensitivity
Fe-16Cr	131	309	29.5	Non
Fe-35Cr	191	451	44.1	Non
Fe-50Cr	552	678	17.1	Non
Type430	324	491	18.0	Non
Type304	200	560	52.0	Non

Table 5 Constant strain rate test results, at a strain rate of $4 \times 10^{-7} \text{sec}^{-1}$ in PWR primary water at 320°C

Stress corrosion cracking occurred only for Type 304 stainless steel in the oxygenated water (simulated BWR coolant environment). No material and test environments except the Type 304 stainless steel in simulated BWR coolant showed SCC sensitivity.

Figure 3 Stress-Strain curves obtained by tensile test in Ar gas

Figure 4 Stress-Strain curves obtained by SSRT in high purity water containing 8ppm DO (simulated BWR cooling water)

It is well known that the SCC path of conventional austenitic stainless steels subjected to sensitization treatment propagates intergranularly, due to Cr-depletion and/or segregation of impurities in materials at grain boundaries. In this work, the behaviour of intergranular SCC for the sensitized type304 stainless steel shows good agreement with the above-mentioned aspect [3-7].

In the CERT/SSRT method, SCC sensitivity is evaluated by the increase in SCC fraction on the fractured surface, and by the decrease in ultimate tensile strength and elongation as compared with those of non-corrosive environments [3-5], [9]. It was found from this experimental study, shown in Tables 2 ~ 5, that there was no difference in either the strength or the elongation of any material with no SCC sensitivity between water and Argon gas environments. On the other hand, both the ultimate tensile strength and total elongation of Type 304 stainless steel can be clearly seen in Figs. 3 and 4.

From the present SCC tests, it was confirmed that high purity Fe-Cr alloys have high SCC resistance in simulated Light Water Reactor coolants. It is also well-known that the mechano-chemical effects related to SCC sensitivity are easily accelerated and evaluated by CERT/SSRT methods [9]. From this viewpoint, the high SCC resistance of high purity Fe-Cr alloys may mainly be due to formation of a tight, thin oxide film, in which Cr will be enriched [5,6]. Detailed examination and analysis of the oxide film formed in the simulated LWR water environments will be conducted for high purity Fe-Cr alloys.

3.3 Fracture Mode

Every high purity Fe-Cr alloy shows high SCC resistance. The reason for these characteristics may be the formation of Cr-rich oxides, which are formed in corrosive aqueous environments [5, 6]. Only Type 304 stainless steel in the simulated BWR water showed intergranular cracking due to SCC.

The high purity Fe-16Cr, -25Cr, and -35 alloys, and also the intermediate purity Fe-30Cr alloy and Type 430 stainless steel were fractured in ductile mode in a dimple pattern for all corrosive and pure Ar environments. The fracture mode of the high purity Fe-50Cr alloy was a mixture of dimple and cleavage-like transgranular cracking. However, no difference in fracture modes of this material between the corrosive and non-corrosive environments was observed.

4. CONCLUSION

High purity Fe-Cr alloys showed high SCC resistance in simulated BWR and PWR primary cooling waters. What is especially surprising is that even Fe-50%Cr alloy shows high SCC resistance and ductility in these corrosive environments.

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