Inconel Alloy 690—A New Corrosion Resistant Material*

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INCONEL Alloy 690 (Ni-30Cr-9Fe-0.02C, wt. %), is a new high-chromium nickel-base alloy, designed to provide a stable austenitic structure combined with resistance to corrosion and oxidation in aggressive industrial environments at elevated temperatures. Extensive laboratory evaluation of Alloy 690 during the past 12 years has demonstrated remarkable resistance to sensitization, and to stress corrosive cracking in chloride environments. In high temperature water, this material resists stress corrosion attack over a wide range of temperatures, oxygen contents in the presence of crevices, and lead contamination. Alloy 690 has been found to release only a negligible amount of material when exposed to high velocity water at elevated temperatures—a fact that may be of major importance in light water reactor technology.

1. Introduction

INCONEL® alloy 690 is a new austenitic nickel-base alloy that was developed to provide excellent corrosion resistance in many demanding high temperature environments. In particular, it shows promise for applications involving high temperature water (e.g., nuclear steam generators) and nitric acid environments (e.g., pickling and nuclear fuel reprocessing).

Preliminary results were presented in a paper by Copson et al. at The Fifth International Congress on Metallic Corrosion in Tokyo, May, 1972 (See page 376 of proceedings).

Alloy 690 is now in full-scale production and has been fabricated into many forms, including sheet, plate, bar, pipe, tube-shells and thin-wall tubing.

This paper brings together some of the available data on the corrosion characteristics of alloy 690. It also provides a brief description of its physical metallurgy and mechanical properties.

2. Structure and Properties

Alloy 690 is a single phase austenitic alloy with a nominal composition of 60% Ni-30% Cr-9% Fe-0.02% C. This composition places it well within the austenite (γ) field at 816°C****, as shown in Figure 1. The α' phase identified in Figure 1 is a chromium-rich phase, very similar to

Fig. 1. The Ni-Cr-Fe phase diagram showing the location of the $\gamma/\gamma' + \alpha'$ solvus from 816° ~ 1260°C.

Table 1. Effect of high temperature exposure on the impact resistance of mill annealed alloy 690

Exposure	Room temperature impact resistance CVN (Joules)
None	189
12,000 h/566°C	164
13,248 h/593°C	169
12,000 h/649°C	172
12,000 h/760°C	184

the iron-chromium σ phase both in morphology and hardness. It can cause embrittlement when precipitated during prolonged high temperature exposure in alloys whose compositions lie in the $\gamma + \alpha'$ field. Since the composition of alloy 690 lies well away from the $\gamma + \alpha'$ field, precipitation of the embrittling α' would not be expected. This is confirmed by the impact properties listed in Table

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**** 816°C is the lowest temperature at which the $\gamma/\gamma' + \alpha'$ solvus has been determined (1).

1. These data show that the toughness of alloy 690 is virtually unaffected by 12,000 hour exposures at temperature in the range 566~760°C. It should be noted that impact resistance is particularly sensitive to precipitation of hard and brittle phases such as α' and σ . For example, room temperature impact resistance for Type 310 stainless steel can drop from ~270J to less than 14J due to the formation of σ phase at ~700°C.

Carbide precipitation can take place in alloy 690 provided the carbon content is high enough. The carbide in alloy 690 is Cr_{23}C_6 . Although carbon solubility has yet to be totally defined, experience indicates that heating to 1150°C will dissolve all the carbide in a 0.04% C material. As a result, sensitization due to chromium depletion is possible on slow cooling. However, if the carbon content is kept at 0.02% or below, alloy 690 cannot be sensitized to the Huey test. Also, levels of 0.02% C can be readily achieved with the argon-oxygen decarburization process used in the refining of alloy 690.

The fully austenitic structure of alloy 690 means that it can be fabricated by the same mill practices used to hot and cold work other commercial high-nickel alloys such as INCONEL® alloy 600. Because of its higher chromium content, alloy 690 work hardens slightly more than alloy 600, as shown in Figure 2. However, this presents no problems in producing heavily worked products such as sheet and thin-wall tubing. The effect of cold work on the room temperature mechanical properties of alloy 690 is shown in Figure 3. Both the strength and ductility respond as expected to

Fig. 2. Work-hardening Alloy 690, Alloy 600 and Nickel 200.

Fig. 3. Effect of cold work on Tensile properties of 12.7 mm hot rolled plate, annealed at 1205°C/1 h/WQ.

Fig. 4. Short-time elevated temperature Tensile properties of 3.8 mm cold rolled strip, annealed at 1038°C.

increasing cold work.

Increasing the tensile test temperature decreases the tensile and yield strengths (Figure 4); however, temperatures in excess of 600°C are needed to cause a significant decrease in strength. At steam generator temperatures of ~316°C, alloy 690 exhibits high strength properties which are insensitive to changes in temperature in this region. A detailed description of creep and stress-rupture properties for alloy 690, is given in Reference 2.

Regarding welding, alloy 690 can be joined using alloy 600 welding products; also a filler of matching composition has been developed for gas tungsten-arc and gas metal-arc welding, as well as a covered electrode. These can be made commercially available when demand warrants it. There is no evidence of any impairment of stress corrosion resistance due to welding with gas tungsten-arc welds prepared with a matching filler.

Table 2. Environments used to evaluate the corrosion behavior of Alloy 690

<u>General corrosion</u>	
HNO ₃ -HF acid solutions	
Deaerated 50% NaOH/316°C	
Undeaerated 50% NaOH/300~332°C	
Deaerated sodium phosphate solutions/275 & 325°C	
Deaerated ammoniated water/316°C/5.5 mps	
Deaerated borated water/316°C/5.5 mps	
<u>Localized corrosion</u>	
Boiling 65% HNO ₃	
Polythionic acid	
Deaerated 500 ppm Cl water/316°C	
<u>Stress corrosion</u>	
Chloride environments	
Boiling MgCl ₂ /154°C	
Undeaerated water + 500 ppm Cl/260°C	
Undeaerated water + 875 ppm Cl/260°C	
Undeaerated water + 660 ppm Cl + 150 ppm Na ₂ HPO ₄ /260°C	
Deaerated (hydrazine) Water + 500 ppm Cl/316°C	
Caustic environments	
Undeaerated 50% NaOH/300°C	
Deaerated 50% NaOH/284, 300, 316, 332°C	
Deaerated 10% NaOH/316°C	
High temperature water	
Undeaerated water + crevice/316°C	
Undeaerated water (36 ppm O)/289°C	
Undeaerated water (36 ppm O → 8 ppm O)/289°C	
Deaerated pH 10 water/316°C and 360°C	
Deaerated pH 10 water + 60 ppm Na ₂ HPO ₄ /326°C	
Deaerated water/316°C	
Deaerated water + Pb/316°C	
Other environments	
pH 2.5 H ₂ SO ₄ at controlled potential	
Steam + 10 ppm Cl + 20 ppm O/280~430°C	

Table 3. Composition of Alloy 690 heats used in corrosion studies

	Chemical composition (wt. %)			
	C	Cr	Fe	Ni
Commercial heats				
Y24A7L	0.02	29.9	8.7	Bal
NX10C1H	0.03	30.8	9.6	"
NX4458H	0.01	27.9	8.8	"
NX20C4H	0.02	30.2	9.0	"
NX4460H	0.06	28.3	8.9	"
Laboratory heats				
T-57177	0.05	29.3	10.1	Bal
T-99016	0.07	29.3	11.4	"
T-53176	0.07	28.8	12.4	"
T-53838	0.07	28.3	12.1	"
T-61540 (Filler)	0.06	29.3	11.7	Bal

3. General Corrosion

As indicated in Table 2, the corrosion resistance of alloy 690 has been evaluated in a number of

Table 4. Corrosion rates of Alloy 690 in various nitric-hydrofluoric acid mixtures

Solution	Corrosion rate*	
	(g/(m ² .d))	(mm/yr)
10% HNO ₃ +3% HF	3.3	0.15
15% HNO ₃ +3% HF	5.6	0.25
20% HNO ₃ +3% HF	3.3	0.15

* Average for duplicate specimens tested at 60°C.

environments known to cause general, localized or stress corrosion attack in other alloys. The compositions of alloy 690 which were investigated are listed in Table 3.

3.1. HNO₃-HF Acid Solutions

The reason for testing in mixed nitric-hydrofluoric acids was to evaluate the potential of alloy 690 as a vessel material for processing spent nuclear fuel elements as well as for the tanks of pickling baths. Because of its high chromium con-

Fig. 5. Descaled weight loss after 2-week exposure to deaerated 50% NaOH at 316°C (duplicate specimens, Alloy 690 from heat NX10C1H).

Fig. 6. Descaled weight loss after exposure to undeaerated 50% NaOH (single specimens, Alloy 690 from heat Y24A7L).

tent, the alloy exhibits excellent resistance to various nitric-hydrofluoric acid mixtures, as shown in Table 4.

3.2. Deaerated 50% NaOH at 316°C

Testing in high temperature 50% NaOH environments helped to establish the corrosion resistance of alloy 690 under conditions simulating

the most severe superheating imaginable on the secondary side of pressurized water reactor (PWR) steam generators. When the temperature of a layer of water at a heat transfer surface exceeds the temperature of the main body of slightly alkaline water by a temperature difference ΔT , a high concentration of NaOH can develop in this layer³⁾. The maximum ΔT attainable on the secondary side of PWR steam generators is not known. The 50% NaOH level selected for testing is equivalent to a ΔT of some 27°C (80°F), which may be higher than that encountered in practice. The 50% NaOH test data, both with regard to general corrosion and stress corrosion cracking, must therefore be regarded as representing a theoretical extreme.

The descalled weight loss of alloy 690 and a number of other alloys after exposure in deaerated 50% NaOH at 316°C is shown in Figure 5. The corrosion scales were removed using the alkaline permanganate-acid method⁴⁾. The results, which are plotted on a logarithmic scale, show that alloy 690 exhibits a corrosion rate intermediate between that of alloy 800 and alloy 600.

3.3. Undeaerated 50% NaOH at 300° and 332°C

Descalled weight loss resulting from exposure of the same alloys to undeaerated 50% NaOH at 300° and 332°C is shown in Figure 6. For these specimens, the weight loss was determined using the electrolytic-sulfuric acid descaling method⁴⁾. Again, the results show that alloy 690 exhibits a corrosion rate between that of alloy 800 and alloy 600.

3.4. Deaerated Sodium Phosphate Solutions at 275° and 325°C

Corrosion test results of alloy 690 and other alloys in high temperature sodium phosphate solutions have been recently published by Pessal et al.⁵⁾ and

Table 5. Corrosion of various alloys in high temperature sodium phosphate solutions

Test environment				Descalled weight loss (mg/dm ² in 1 yr)			
Na/P	Concentration (Molality)	Temp. (°C)	Maximum exposure time (h)	Alloy 600	Alloy 690	Alloy 800	304 SS
1.35	6.0	325	315	680	1600	620	2139
1.6	6.0	325	480	2800	5600	6400	5989
2.0	5.0	325	504	510	600	600	599
2.2	0.18	325	504	86	43	43	257
2.3	5.0	325	451	470	471	1200	11123
1.6	6.0	275	1344	2400	—	—	3000
2.3	5.0	275	1226	86	150	260	—
2.3	0.4	275	1560	540	—	340	—
2.6	0.15	275	1176	43	1000	100	—

Table 6. High velocity loop test parameters

Parameter	Borated water	Ammoniated water
Temperature	316°C	316°C
Pressure	13.8 MPa (2000 psig)	13.8 MPa (2000 psig)
Flow Rate	5.5 m/s (18 ft/s)	5.5 m/s (18 ft/s)
Hydrogen	25 cc (STP/kg H ₂ O)	25 cc (STP/kg H ₂ O)
Oxygen	0~15 ppb	0~15 ppb
Boron (H ₃ BO ₃)	1500 ppm	
Lithium (LiOH)	0.7 ppm	
pH	5.5	9.5~10.9*
Volume of Loop	32.3 l (8.5 gal)	32.3 l (8.5 gal)

* Adjusted with ammonia.

are reproduced in part in Table 5. The extent of attack on the various alloys is dependent on the phosphate concentration and the Na/P ratios, with the higher nickel alloys generally exhibiting greater resistance to attack than Type 304 stainless steel.

3.5. Deaerated Ammoniated and Borated Water at 316°C

In pressurized water reactors, a factor of particular interest is the corrosion resistance of steam generator tubing materials in flowing primary water. Material lost to the stream can become radioactive by coming in contact with the reactor core and then redeposit on the primary circuit surfaces. A recent evaluation of the corrosion behavior of alloy 600 and alloy 800 in a CANDU reactor outlet autoclave during pressurized lithiated operation has shown that these two alloys exhibit comparable corrosion resistance⁶⁾. The corrosion resistance of alloy 690 under these and similar primary coolant conditions is not known. In an attempt to obtain some insight into how alloy 690 is likely to behave, a laboratory test program was carried out employing a high velocity test loop. Alloy 690 was evaluated in two simulated primary water environments, referred to here as "ammoniated" and "borated". The test parameters in these environments are shown in Table 6.

The alloy 690 test specimens and those of control materials were heat treated for 0.5 h at 980°C/AC. The surfaces were prepared by grinding on a wet 120 grit silicon carbide belt to a 0.75 μ m finish. The specimens were weighed and then exposed for extended periods of time under the conditions shown in Table 6. After exposure, the specimens from the ammoniated water tests were descaled by cathodic charging in 5% H₂SO₄ inhibited with quinoline ethiodide⁴⁾. The specimens from the borated water tests were descaled by the alkaline permanganate acid method⁴⁾. The method of reporting results and terminology adopted are

defined in NACE standard TM-02-74, covering the following terms:

1. "Descaled metal loss" (metal consumed): The difference between initial weight and weight after removal of adherent corrosion film.
2. "Corrosion film weight" (adherent corrosion film): The difference between the weight before descaling and the weight after descaling.

The difference between the descaled metal loss and the corrosion film weight represents material

Fig. 7. Effect of chromium content on material lost to stream in 316°C deaerated water flowing at a velocity of 5.5 m/s. Alloy 690 from Heat Y24A7L.

lost to the stream. Alloys showing the least loss of material to the stream would be expected to produce less activity on the primary water side of steam generators.

The behavior of alloy 690 in ammoniated and borated water is shown in Figure 7. It can be seen that alloy 690 loses less material to the stream than any of the other materials tested. The films formed on alloy 690 as a result of the ammoniated and borated water exposures were of the thin tarnish type and appeared to be extremely adherent to the metal.

The results suggest that the crud release rates of alloy 690 in an operating steam generator could be very low. The use of this alloy in steam generators could therefore reduce the amount of activity built up on the primary side. This is assuming that an important source of the active species is the material being corroded from the tube wall. A note of caution is warranted here; the data shown in Figure 7 are subject to a standard error of about 25% and should be regarded as a guide rather than a precise measurement.

4. Localized Corrosion

4.1. Intergranular Corrosion Tests in Boiling 65% HNO₃

The intergranular corrosion resistance of alloy 690 was evaluated in boiling 65% HNO₃. This test, known as the Huey test, is standardized as ASTM A262, Practice C. It is a very severe test which for stainless steels attacks chromium depleted areas, carbides, and σ , when present.

The intergranular corrosion resistance of alloy

690 determined by the Huey test was found to be critically dependent on the carbon content of the alloy. As shown in Figure 8, alloy 690 containing 0.02% C shows very low corrosion rates in boiling

Fig. 8. The effect of carbon content on the corrosion rate of Alloy 690 in the huey test. Material annealed 1 h/1150°C/WQ before sensitizing.

Table 7. Corrosion rates of Alloy 690 welded with matching filler in boiling 65% nitric acid

Heat No.	% C	Corrosion rate	
		(g/m ² .d)	(ipm)
NX20C4H	0.02	2	0.0003
NX10C1H	0.03	2	0.0003

Fig. 9. Time-temperature-sensitization diagram for Alloy 690 in boiling 65% Nitric acid. Heat T-57177 containing 0.05% c, solution annealed 1 h/1150°C/WQ.

Table 8. Polythionic acid and nitric acid test results

Alloy	Heat treatment	Polythionic acid test ^{a)}	Corrosion rate ^{b)} in boiling 65% HNO ₃	
		No. of specimens cracked/number destructively examined	(g/(m ² .d))	(mm/yr)
690*	0.5 h @ 1038°C/AC	0/2	3.3	0.15
	0.5 h @ 1038°C/AC			
	+100 h @ 316°C/AC	0/2	2.0	0.09
	0.5 h @ 1038°C/AC			
304SS	+1000 h @ 316°C/AC	0/2	3.3	0.15
	1 h @ 1065°C/WQ			
	+2 h @ 675°C/AC	1/1 ^{c)}	—	—

^{a)} Test duration=720 h; u-bend test specimens

^{b)} Average of five 48-h periods

^{c)} Cracks within 1 h, qualifying test

* Heat Y24A7L

65% HNO₃, irrespective of heat treatment. For the 0.02 and 0.03% C alloys, the corrosion rates represent the average values of five consecutive 48-hour exposures, whereas for the 0.05 and 0.07% C alloys, the weight loss is an average of two 48-hour exposure periods. The extremely low corrosion rate of the 0.02% C alloy, together with the fact that no detectable intergranular attack could be detected by conventional metallographic examination of sections after corrosion testing, suggest that the 0.02% C alloy is resistant to sensitization.

Alloy 690 containing 0.02 and 0.03% C was also examined in the welded condition. Test coupons, 0.5 cm thick, were butt welded using the matching filler and exposed for five 48-hour test periods to boiling 65% HNO₃. Again, as shown in Table 7, very low rates of corrosion were found, with the only attack observed being light interdendritic etching of the matching filler.

Higher carbon versions (e.g., 0.05% C) of alloy 690 can be furnace sensitized to give significant intergranular attack in boiling nitric acid, as shown in the time-temperature-sensitization diagram in Figure 9. Effects of welding on the sensitization behavior of such higher carbon materials still need to be evaluated.

4.2. Polythionic Acid Tests

In conjunction with the boiling nitric acid tests, alloy 690 was also evaluated for resistance to polythionic acid cracking. The standard polythionic acid test was employed, with the potency of the acid solution being demonstrated by cracking a sensitized type 304 stainless steel within 1 hour. Alloy 690 exhibited complete resistance to cracking in this solution, both in the annealed condition and after prolonged aging at 316°C (Table 8).

4.3. Pitting Tests in Chloride Solutions

Alloy 690 specimens from heats NX10C1H, NX4460H, and NX4458H (see Table 3) were exposed for 96 weeks at 316°C in water containing 500 ppm chloride, and deaerated with N₂H₄, to determine whether pitting would occur. No evidence of pitting or any other type of attack was found using optical and scanning electron microscopes at magnifications up to 3000X.

5. Stress Corrosion Cracking

5.1. Chloride Environments

Alloy 690 has been tested for resistance to chloride stress corrosion cracking in a wide variety of high temperature chloride environments. The controls used in these tests were alloy 800 and Type 304 stainless steel. Both of these alloys are known to crack in elevated temperature chloride environments, with alloy 800 being more resistant than Type 304 stainless steel. In all cases, alloy 690 was tested for periods significantly in excess of those required to crack alloy 800. The following tests were employed:

1. Boiling MgCl₂, 154°C, 13.9 weeks.
2. Undeaerated water + 500 ppm chloride (NaCl), 260°C, 18 weeks.
3. Vapor phase above environment No. 2, 8 weeks.
4. Undeaerated water + 875 ppm chloride (NaCl), 260°C, 8 weeks.
5. Vapor phase above environment No. 4, 8 weeks.
6. Undeaerated water + 660 ppm chloride (NaCl) + 150 ppm Na₂HPO₄, 260°C, 8 weeks.
7. Deaerated N₂H₄ water + 500 ppm chlo-

Table 9. Stress corrosion tests at 260°C in undeaerated water containing 500 ppm chloride (NaCl) to evaluate the effects of heat treatment, cold work, welding and crevices

Alloy	Heat treatment code	Number of specimens (No. of heats)	Test duration (weeks)	Number of specimens cracked/number destructively examined	Maximum depth of attack (mm)
690*	MA	2 (1)	18	0/2	0
	MA+L	2 (1)	18	0/2	0
	MA+A ₁	2 (1)	18	0/2	0
	MA+CR	2 (1)	18	0/2	0
	A ₁ +L	2 (1)	18	0/2	0
	A ₃ +L	2 (1)	18	0/2	0
	A ₂ +W (12.7)	2 (1)	8	0/2	0
	A ₂ +W (25.4)	2 (1)	8	0/2	0
800	A ₄ +L ₀	2 (1)	1	2/2	2.5
304 SS	A ₄ +L ₀	2 (1)	1	2/2	3.0

Double U-bend (crevice) test specimens for alloy 690

Single U-bend test specimens for alloy 800 and 304 SS

Code: MA=Mill Annealed

CR=Cold Rolled 40%

A₁=1 h @ 1093°C/WQ

A₂=1 h @ 1150°C/WQ

A₃=1 h @ 1205°C/WQ

A₄=1 h @ 1065°C/WQ

L₀=5 h @ 704°C/AC

L=2 h @ 650°C/AC

W (thickness, mm)=manual gas tungsten-arc welded at indicated thickness with matching filler

* Heat Y24A7L

ride (NaCl), 316°C, 96 weeks.

In tests using undeaerated water plus chloride or chloride plus phosphate, the solution was prepared by dissolving NaCl in distilled-deionized water. The pH was not adjusted and the autoclave was sealed with 1 atmosphere of air in the headspace. The oxygen content of the solution at temperature could not be measured, but was estimated to be at least 6 ppm at the start of each test period. Specimens were either immersed in the solution or suspended in the vapor phase above the solution.

In the test using deaerated water plus chloride, the solution was prepared by dissolving NaCl in distilled-deionized water to which 65% N₂H₄ was added (0.2 ml of N₂H₄ per 1.6 l of solution). The solution was then deaerated within the closed autoclave by three alternate pressurizing (3.5 MPa-argon)/aspirating (5.7 KPa) cycles, and the autoclave sealed with 1 atmosphere of argon in the headspace. The oxygen content after deaeration was <20 ppb.

Alloy 690 did not exhibit stress corrosion cracking in any of the environments used. Furthermore in Test No. 2 (Table 9), extensive evaluation of the effects of heat treatment, cold work, welding,

and the presence of crevices did not reveal any detrimental variable. Since no evidence of any stress corrosion susceptibility was found, alloy 690 must be regarded as highly resistant to chloride cracking.

5.2. Caustic Environments

Alloy 690 has been tested for resistance to caustic stress corrosion cracking in both undeaerated and deaerated NaOH solutions. The controls used in the caustic tests were Type 304 stainless steel, alloy 800, alloy 600 and commercially pure nickel, Nickel 201. The latter was included when it was realized that all commercially available Fe-Cr-Ni alloys exhibit some degree of cracking in high temperature caustic environments.

The following test environments were employed:

1. Undeaerated 50% NaOH, 300°C.
2. Deaerated 50% NaOH, 284, 300, 316 and 332°C.
3. Deaerated 10% NaOH, 316°C.

For these caustic tests, the autoclave solution was prepared by dissolving ACS certified reagent grade electrolytic NaOH pellets in distilled water. For tests in undeaerated NaOH, the autoclave was sealed with an 0.35 MPa over-pressure of air in

Table 10. Stress corrosion tests in undeaerated 50% NaOH at 300°C to evaluate the effects of heat treatment, cold work, and welding

Alloy	Heat treatment code	Number of specimens (Number of heats)	Test duration (wk)	Number of specimens cracked/number destructively examined	Maximum depth of attack (mm)
690*	MA	2 (1)	3.9	0/2	0
	MA+L	2 (1)	3.9	0/1	0
	MA+A ₁	2 (1)	3.9	0/2	0
	MA+CR	1 (1)	1.7	1/1	2.79
		1 (1)	3.9	0/1	0
	A ₁ +L	2 (1)	3.9	0/2	0
	A ₃ +L	2 (1)	3.9	0/2	0
	A ₂ +W (12.7)	2 (1)	3.9	0/2	0
	A ₃ +W (25.4)	2 (1)	3.9	0/2	0
800	A ₁ +L	2 (1)	1.7	2/2	1.06
	MA	2 (1)	1.3	2/2	2.15
600	CR+A ₁	1 (1)	2.6	1/1	1.06
		1 (1)	3.9	1/1	0.06
	MA+CR	2 (1)	3.9	0/2	0
	A ₁ +L	2 (1)	3.9	2/2	0.02
	A ₃ +L	2 (1)	3.9	2/2	0.06
	A ₁	2 (1)	0.4	2/2	3.05
304 SS	A ₁ +L	1 (1)	0.4	1/1	3.05
	MA	2 (1)	3.9	0/2	0

U-bend test specimens

Code: MA=mill annealed

CR=cold rolled 40%

A₁=1 h @ 1093°C/WQA₂=1 h @ 1150°C/WQA₃=1 h @ 1205°C/WQ

L=2 h @ 650°C/AC

W (thickness, mm)=manual gas tungsten-arc welded at indicated thickness with matching filler

* Heat Y24A7L

the headspace. The specimens were examined after each 3-day exposure period, with a fresh solution being used for each subsequent period. For tests in deaerated NaOH, deaeration was accomplished by three alternate pressurizing (3.5 MPa-argon)/aspirating (5.7 KPa) cycles, followed by a 2-hour argon bubbling treatment. The autoclave was sealed with 1 atmosphere of argon in the headspace.

The results from the undeaerated 50% NaOH tests are shown in Table 10. With the exception of one of two cold rolled specimens, alloy 690 did not exhibit cracking in the 27-day test period, irrespective of heat treatment, cold work, or presence of welds.

The results from the deaerated 50% NaOH tests have been previously reported^{7,8)}. At 316°C, alloy 690 appears to have a slightly lower resistance to stress corrosion cracking than alloy 600 in this environment (see scatterband shown in

Figure 10). However, the differences are small and dependent on test temperature⁷⁾.

In deaerated 50% NaOH, alloy 600 appears to be somewhat more resistant to stress corrosion cracking than alloy 690. The reverse is true in deaerated 10% NaOH, as shown in Figure 10. Here, with the exception of Nickel 201, alloy 690 was the most resistant alloy tested, although again the difference between alloy 600 and alloy 690 is slight.

The caustic stress corrosion test data can be summarized as indicating that alloy 690 is the most resistant of the alloys tested in undeaerated 50% NaOH and deaerated 10% NaOH, while its resistance is slightly inferior to that of alloy 600 in deaerated 50% NaOH.

5.3. High Temperature Water Containing Oxygen

It has been known for a long time⁹⁾ that most commercially available Fe-Cr-Ni alloys exhibit

Fig. 10. Stress corrosion cracking of U-bend specimens in deaerated 10% NaOH at 316°C. Exposure time=6 weeks.

stress corrosion cracking in oxygen-containing (unde-aerated) water at elevated temperatures in the presence of a crevice. In fact, stress corrosion resistance under these conditions was a major parameter in the development of the composition of alloy 690. The "oxygen-plus-crevice" test used here employs double U-bend specimens, with the crevice being formed between the inner and outer members of the specimen. Cracking, when it occurs, is confined to the outer surface of the inner U-bend member, having been initiated within the crevice. In these tests, the environment was distilled and deionized water adjusted to pH 10 with NH_4OH . The autoclave was sealed with 1 atmosphere of air in the headspace, which provides a minimum dissolved oxygen content of 6 ppm.

As shown in Table 11, cracking was readily obtained in the control alloys, whereas alloy 690 survived the 48-week test period without cracking. The cracking resistance of alloy 690 in this environment was not impaired by various heat treatments, cold work, or the presence of welds.

Table 11. Stress corrosion tests in unde-aerated water at 316°C to evaluate the role of heat treatment, cold work, and welding on the "oxygen-plus-crevice" effect

Alloy	Heat treatment code	Number of specimens (No. of Heats)	Test duration (Weeks)	Number of specimens cracked/number destructively examined	Maximum depth of attack (mm)
690*	MA	2 (1)	48	0/2	0
	MA+L	2 (1)	48	0/2	0
	MA+A ₁	2 (1)	48	0/2	0
	MA+CR	2 (1)	48	0/2	0
	A ₁ +L	2 (1)	48	0/2	0
	A ₃ +L	2 (1)	48	0/2	0
	A ₂ +W (12.7)	4 (1)	48	0/4	0
	A ₂ +W (25.4)	2 (1)	48	0/2	0
	MA+W (3.8)+L ₁	2 (1)	48	0/2	0
600	A ₁ +L ₂	2 (1)	2	2/2	2.6
800	A ₁ +L ₂	2 (1)	4	2/2	2.0
304 SS	A ₁ +L ₂	2 (1)	4	2/2	3.0

Double U-Bend (crevice) test specimens

Code: MA=Mill Annealed

CR=Cold Rolled 40%

A₁=1 h @ 1093°C/WQ

A₂=1 h @ 1150°C/WQ

A₃=1 h @ 1205°C/WQ

L=2 h @ 650°C/AC

L₁=4 h @ 593°C/AC

L₂=5 h @ 704°C/AC

W (thickness, mm)=manual gas tungsten-arc welded at indicated thickness with matching filler

* Heat Y24A7L

Table 12. Stress corrosion tests of Alloy 690 of single and double U-bends in highly oxygenated 289°C water

Heat	Heat treatment of outer U-bend member	Heat treatment of inner U-bend member	Number of specimens cracked/number destructively examined	Result
NX10CIH	A	MA+W (12.7)	0/1	No attack
	A	MA+W (12.7)	0/1	No attack
	A+L	None tested	0/2	No attack
Y24A7L	A	MA+W (12.7)	0/1	Weld defect
	A	MA+W (12.7)	0/1	No attack
	MA	None tested	0/2	No attack
Both	MA (Y24A7L)	MA+CR (NX10CIH)	0/1	No attack
	MA (Y24A7L)	MA+CR (NX10CIH)	0/1	No attack

Code: MA=Mill annealed

A=1 h @ 1120°C/WQ

L=2 h @ 675°C/AC

W (thickness, mm)=manual gas tungsten-arc welded at indicated thickness with matching filler.

CR=Cold rolled 40%

Exposure: 47.3 weeks in water containing 36 ppm oxygen, followed by 20.6 weeks in water containing 8 ppm oxygen.

Studies by Clarke et al.¹⁰⁾ suggest that alloy 690 is not immune to cracking in very high oxygen environments (i.e., 36 ppm oxygen) at 289°C. However, the microstructure of the material tested was not typical of wrought alloy 690. A subsequent series of tests repeated by Clarke and McIlree¹¹⁾ using material with well defined heat treatments, did not reveal cracking, although one of the welded specimens had a defect in the filler material. It could not be ascertained whether this defect was a pre-existing shrinkage void or had been caused by the exposure to the environment. Since previous tests had not indicated any susceptibility to cracking by the filler, it seems likely that the observed defect was due to shrinkage during welding. The results of these stress corrosion tests are shown in Table 12.

The stress corrosion tests in oxygen containing water in the presence of crevices suggest that alloy 690 is highly resistant to cracking under these conditions. However, in view of the findings by Clarke¹⁰⁾, alloy 690 should not be regarded as immune in oxygen-saturated high temperature solutions. It seems extremely unlikely, however, that oxygen saturated solutions would ever be encountered in operating boiling water (BWR) or pressurized water (PWR) reactors.

5.4. High Temperature Water with Low Oxygen Content

Alloy 690 has been extensively tested for resistance to stress corrosion cracking in high temperature low oxygen water using alloy 600 as the control. The following test environments were

employed:

1. Deaerated pH 10 water, 316°C (48 weeks) and 360°C (60 weeks).
2. Deaerated pH 10 water + 60 ppm Na_2HPO_4 , 326°C, 96 weeks.
3. Deaerated water, unadjusted pH, 316°C, 48 weeks.
4. Deaerated water, unadjusted pH + Pb, 316°C, 18 weeks.

For the 316°C tests in deaerated pH 10 water, the solution was prepared by deaerating distilled-deionized water (resistivity $>1 \text{ M}\Omega\text{-cm}$) with argon in the closed autoclave by three alternate pressurizing/aspirating cycles. This was followed by bubbling with argon for 2 hours. The autoclave was sealed with 1 atmosphere of argon in the headspace. This procedure gives an oxygen content of about 20 ppb. The pH was adjusted with NH_4OH .

The 360°C tests in deaerated pH 10 water were carried out using a refreshed autoclave. The test solution was distilled-deionized water adjusted to pH 10 with NH_4OH and deaerated with hydrogen gas and catalyzed N_2H_4 (35% N_2H_4 with organic catalyst). The water was pumped from a 190 l (50 gal) holding tank to the autoclave at a rate of 1.14 l/h (0.3 gal/h). This procedure again yields oxygen contents of <20 ppb.

For tests in deaerated pH 10 water with phosphate, deaeration was carried out by bubbling with nitrogen for 3.5 hours and addition of 0.65 ml/l of hydrazine ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$). Sodium hydroxide was used to adjust the pH to 10. This procedure

Table 13. Stress corrosion tests in deaerated pH 10 water at 316°C to evaluate the effects of heat treatment, cold work, welding and crevices

Alloy	Heat treatment code	Number of specimens (Number of heats)	Test duration (wk)	Number of specimens cracked/number destructively examined	Maximum depth of attack (mm)
690*	MA	2 (1)	48	0/2	0
	MA+L	2 (1)	48	0/2	0
	MA+A ₁	2 (1)	48	0/2	0
	CR	2 (1)	48	0/2	0
	A ₁ +L	2 (1)	48	0/2	0
	A ₃ +L	2 (1)	48	0/2	0
	A ₂ +W (12.7)	2 (1)	18	0/2	0
	A ₂ +W (25.4)	2 (1)	18	0/2	0
600	CR+A ₁	2 (1)	48	0/2	0
	MA+CR	2 (1)	18	1/2	2.79
		2 (1)	48	0/2	0
	A ₁ +L	2 (1)	48	0/2	0
	A ₃ +L	2 (1)	48	0/2	0

Double u-bend (crevice) test specimens

Code: MA=mill annealed

CR=cold rolled 40%

A₁=1 h @ 1093°C/WQA₂=1 h @ 1150°C/WQA₃=1 h @ 1205°C/WQ

L=2 h @ 650°C/AC

W (thickness, mm)=manual gas tungsten-arc welded at indicated thickness with matching filler

* Heat Y24A7L

Table 14. Stress corrosion tests in deaerated 316°C water (unadjusted pH) to evaluate the effects of heat treatment, welding and the presence of crevices

Alloy	Heat treatment code	Number of specimens (Number of heats)	Test duration (wk)	Number of specimens cracked/number destructively examined	Maximum depth of attack (mm)
690*	MA	4 (1)	48	0/4	0
	MA+L	4 (1)	48	0/4	0
	A ₁	4 (1)	48	0/4	0
	A ₂	6 (1)	48	0/6	0
	A ₁ +L	4 (1)	48	0/4	0
	A ₃ +L	4 (1)	48	0/4	0
	CR	4 (1)	48	0/4	0
	A ₂ +W (12.7)	4 (1)	48	0/4	0
600	A ₂ +W (25.4)	2 (1)	48	0/2	0
	MA	4 (1)	48	1/4	0.05
	A ₁	4 (1)	48	1/4	0.12
	A ₁ +L	4 (1)	48	0/4	0

Double u-bend (crevice) test specimens

Code: MA=mill annealed

CR=cold rolled 40%

A₁=1 h @ 1093°C/WQA₂=1 h @ 1150°C/WQA₃=1 h @ 1205°C/WQ

L=2 h @ 650°C/AC

W (thickness, mm)=manual gas tungsten-arc welded at indicated thickness with matching filler

* Heat Y24A7L

Table 15. Supplementary higher temperature (360°C) stress corrosion tests of Alloy 690*
U-bends in deaerated pH 10 water to evaluate the effects of heat treatment

Heat treatment code	Number of specimens (Number of heats)	Test duration (wk)	Number of specimens cracked/number destructively examined	Maximum depth of attack (mm)
M	4 (2)	60	0/4	0
A ₄	2 (1)	60	0/2	0
A ₄ +L ₄	2 (1)	60	0/2	0

Code: M=0.5 h @ 1010°C/AC

A₄=1 h @ 1120°C/WQ

L₄=1 h @ 675°C/AC

* Heats NX 4458H, NX 4460H

gives an oxygen content of 10~20 ppb.

Solutions with unadjusted pH were prepared by pressurizing/aspirating with argon followed by steaming some of the solution from the autoclave. This procedure yields oxygen contents between 0 and 5 ppb. In the preparation of the unadjusted pH solutions containing lead added as an impurity (10 g lead powder/1.8 l of water), the steaming step was omitted, and the autoclave was sealed with one atmosphere of argon in the headspace. This technique yields an oxygen content of about 20 ppb.

Alloy 690 resisted cracking in all the deaerated water tests employed. In deaerated water tests No. 1 and No. 3, a wide range of heat treatments, cold work, welding, and the presence of crevices were evaluated. As shown in Tables 13 and 14, none of these variables appeared to impair the stress corrosion cracking resistance of alloy 690. Also, in test No. 2, increasing the test temperature to 360°C and the exposure time to 60 weeks did not produce cracking in alloy 690 (Table 15).

These results can be summarized as showing that alloy 690 does not appear to be susceptible to stress corrosion cracking in the low oxygen waters which Coriou et al.¹²⁾ and Copson et al.^{9),10)} have shown to be capable of producing intergranular penetration in other high nickel alloys.

5.5. Other Environments

In addition to the stress corrosion tests of alloy 690 already described, there have been two other evaluations carried out. In a study by Vermilyea¹⁴⁾, alloy 690 was tested at a controlled electrochemical potential of 0 volts (standard hydrogen electrode) in a pH 2.5 H₂SO₄ solution at 289°C. In the other, Hammond et al.¹⁵⁾ evaluated alloy 690 in a steam environment containing 10 ppm chloride and 20 ppm oxygen, with the temperature being cycled between 280 and 430°C. Alloy 690 exhibited com-

plete resistance to stress corrosion cracking in both of these environments.^{14,15)}

6. Summary

INCONEL alloy 690 is a stable austenitic high-chromium nickel-base alloy which represents a useful addition to the corrosion resistant Ni-Cr-Fe alloy family. Its high chromium content ensures a high corrosion resistance in oxidizing acids and in flowing high temperature water environments, while its high nickel content provides a high resistance to chloride stress corrosion cracking. The low carbon content of alloy 690 prevents sensitization by chromium depletion in the vicinity of precipitated grain boundary carbides. The alloy appears to be highly resistant to stress corrosion cracking in various aqueous environments at temperatures encountered in nuclear steam generators.

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