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Corrosion Resistance of INCONEL Alloy 617 in Simulated Gas Turbine Environments

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ABSTRACT

It is known that the efficiency of a gas turbine engine can be improved substantially by increasing the operating temperature. However, increasing the temperature puts a tremendous demand on the performance of materials of construction such as the combustion chamber, transition ducts etc. The material has to be resistant to long-term isothermal and cyclic oxidation conditions. In addition, the thermal stability of the alloy is of utmost importance. INCONEL® alloy 617 is a solid solution, nickel - chromium - cobalt - molybdenum alloy with an exceptional combination of high-temperature strength and oxidation and carburization resistance. In this paper, the performance of alloy 617 in isothermal and cyclic oxidation, carburization and burner rig oxidation/sulfidation environments will be presented. In addition some mechanical properties data will be included to show the stability of the alloy after exposure to high temperatures for extended periods of time. For comparison the data obtained on such alloys as INCO® alloy HX and Haynes® alloys 188 and 230 will also be reported.

INTRODUCTION

INCONEL alloy 617 is a solid solution strengthened nickel-chromium-cobalt-molybdenum alloy containing about 1.2% aluminum. Nickel and chromium provide the alloy with resistance to various oxidizing and reducing environments. Cobalt and molybdenum provide the solid solution strengthening. Chromium and aluminum provide the oxidation and carburization resistance. The alloy combines exceptionally high strength with oxidation resistance and long-term thermal stability. Alloy 617 is therefore used as a material for such components as ducting, combustion cans, and transition liners in both aircraft and land based gas turbines. In this paper the performance of alloy 617 in isothermal and cyclic oxidation environments, as well as carburization and burner rig oxidation/sulfidation environments will be presented. Some of the results obtained on other alloys such as alloys 230, HX and 188 are also presented. In addition the effect of long-term exposure on the microstructure of alloy 617 and other alloys will also be presented.

EXPERIMENTAL PROCEDURES

MATERIALS

The alloys of this study were obtained from their commercial producers. Alloy 617 was obtained in the form of 16 mm (0.625") diameter rod from which 7.6 mm (0.3") diameter x 19 mm (0.75") length pins were machined. Other alloys included in the study were INCO alloy HX, HAYNES alloys 188, and 230. Some of the samples were also in the form of 5 mm (0.20") thick sheets about 19 mm (0.75") in length. The chemical compositions of alloys used in this study are reported in Table 1. Although the results are presented for various alloys, the emphasis will be placed on alloys 617, 230, 188 and HX as they are often considered for gas turbine applications. The alloys were exposed to air plus 5 and 20% moisture isothermal oxidation tests. Tests were also conducted in cyclic oxidation, carburization and burner rig oxidation/sulfidation environments. The experimental procedure for each test is described below.

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Oxidation Test Procedure

Samples of alloys in the form of pins mentioned above were exposed to air plus 5 and 20% water vapor for about 1000 hours at 1000°C (1832°F). The moisture was added by bubbling air through water maintained at 33°C and 60.4°C (91°F and (141°F)to obtain 5 and 20% water vapor respectively in the environment. If natural gas is used as a fuel in the combustor of a gas turbine, one of the products of combustion is water and therefore a study of the performance of the alloys in air containing 20% water vapor is reasonable. The oxidation experiments were carried out in an electrically heated 100 mm (3.9") mullite tube. Samples were placed in cordierite boats. In order to obtain mass change data with respect to time the experiment was stopped periodically and samples were allowed to cool to room temperature and then weighed.

Table 1

Nominal Compositions of Alloys in This Study

Alloy	C	Ni	Mo	Si	Cr	Fe	Al	Co	Other
617	0.08	Bal	9.5	0.13	22.0	1.5	1.2	12.5	0.3 Ti
230	0.10	Bal	3.0	0.50	22.0	3.0*	0.2	3.0*	14.0 W
HX	0.10	Bal	9.0	0.20	22.0	19.0	-	1.5	
188	0.01	22.0	-	-	22.0	3.0	-	Bal	14.0 W
86	0.05	Bal	10.0	0.20	25.0	5.0	-	2.0	0.03 Ce
S	0.02	Bal	14.5	0.40	15.5	1.0	0.20	-	0.02 La
214	0.02	Bal	-	-	16.0	2.5	4.35	-	0.01 Y
625	0.05	Bal	9.0	0.20	21.5	2.5	0.2	-	0.2 Ti
C-276	0.02	Bal	16.0	0.08*	15.5	5.0	-	2.5*	4.0 W
601	0.02	Bal	-	0.12	22.5	13.6	1.3	-	0.3 Ti
600	0.05	Bal	-	0.21	16.0	7.0	0.15	-	0.2 Ti
800	0.04	Bal	-	0.35	21.4	Bal	0.35	-	0.4
330	0.06	34.8	-	1.30	19.2	Bal	-	-	1.75 Mn
DS	0.03	34.3	-	2.20	18.0	Bal	-	-	1.30 Mn
309	0.08*	13.5	-	1.0*	23.0	Bal	-	-	2.00 Mn

*** Maximum**

In the cyclic oxidation environments, samples were exposed for 15 minutes at temperature and 5 minutes in cooling air. The experiments were conducted at 1093°C (2000°F).

Oxygen-Sulfur Dioxide Test Procedure

In this experiment a mixture of 96% O_2 and 4% SO_2 was used and the samples were exposed for 1008 h at 704°C (1300°F) in a similar setup as that for oxidation experiments. To obtain mass change data with time the test was again interrupted for weighing. However, in this case, to minimize oxidation in air during thermal cycling, the samples were moved from the hot zone of the furnace to the cold end under argon by means of a pusher rod without breaking the atmosphere seal of the tube. The specimens were removed from the furnace after cooling for a minimum of 1 hour.

Carburization Test Procedure

Carburization testing was conducted in two environments namely H_2-1%CH_4 (carbon activity of 1) and H_2-5.5%CH_4-4.5%CO_2 (partial pressure of O_2 was $10^{-20.57}$ atmospheres with a carbon activity of 1) at 1000°C (1832°F). The samples were in the form of pins similar to those used in the oxidation test. The samples were removed periodically for weighing by pulling the samples to the cold end of the test chamber which was purged with argon to minimize oxidation. Additional details of the test can be obtained from reference 1.

Burner Rig Test

The resistance of gas turbine alloys to sulfidation is normally tested in a burner rig that burns fuel used in the gas turbines. The fuels contain a certain amount of sulfur or sometimes more sulfur could be added externally. In this case the sulfur was limited to that normally contained in JP-5 fuel. Details of the test procedure and a schematic of the burner-rig are reported elsewhere (1,2). The burner-rig tests were conducted at temperatures of 927°C and 1093°C (1700°F and 2000°F). Samples were in the form of pins 3.2 mm (0.125") dia and 38.1 mm (1.5") in length. Samples are loaded in a rotating disc which is placed in the path of the flame for 58 minutes and cooling air for 2 minutes.

Microstructural Stability

To evaluate the microstructural stability of alloys after long term exposure to intermediate temperatures samples were exposed to temperatures varying from 649 to 982°C (1200 to 1800°F) for 100 and 500 hours. After exposure the samples were tested for their impact strength. Full size specimens were used. Sections from some of the impact tested samples were mounted for metallography. Another half of the sample was used for X-ray diffraction analysis of the residue present in the sample after dissolving the matrix by electrolysis in 10% HCl-methanol solution.

RESULTS AND DISCUSSION

Oxidation Test

Figure 1 shows the mass change in air plus 5% water vapor for various commercial alloys including alloys 617 and 230. The samples were exposed at 1000°C (1832°F) for up to 1000 hours. The data indicates that both alloys 617 and 230 have comparable oxidation resistance at 1000°C. The mass change data in air plus 20% water vapor for various alloys are shown in Figure 2. However in this case alloy 617 appears to show much better resistance to scale spallation than all the alloys shown in Figure 2 and in particular alloys 230, HX and 188.

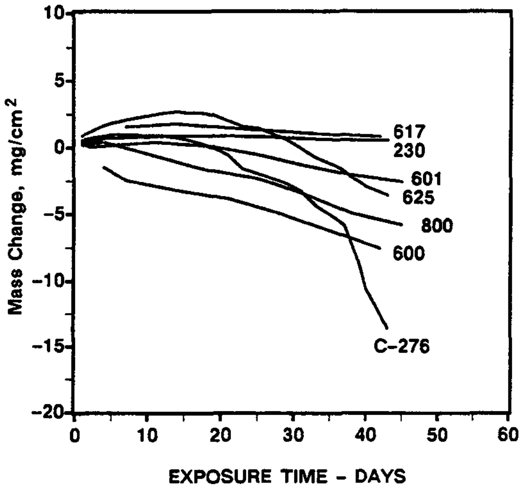

Figure 1. Mass Change Versus Exposure Time in Air Plus 5% Water Vapor at 1000°C.

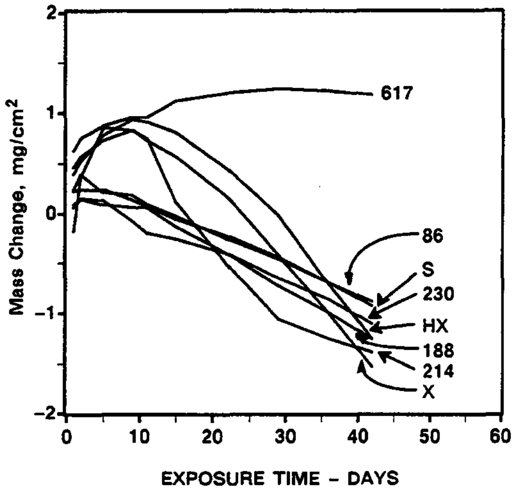

Figure 2. Mass Change Versus Exposure Time in Air Plus 20% Water Vapor at 1000°C.

Figure 3 shows the mass change with respect to number of cycles for alloys 230 and 617 in cyclic oxidation at 1093°C. Here the samples were heated to temperature in 15 minutes and cooled in air for 5 minutes. Samples were weighed after every 100 cycles to obtain the mass change. The final mass change data is shown in Table 2 for samples tested at 1093°C where alloy 617 is almost twice as good as alloy 230.

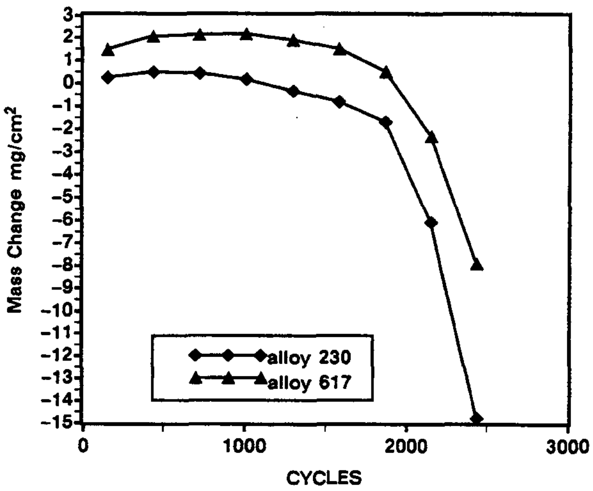

Figure 3. Mass change in cyclic oxidation test at 1093°C versus exposure time for alloys 230 and 617.

Table 2

Mass Change in Cyclic Oxidation Test at 1093°C (The Samples were Heated to Temperature in 15 min. and Cooled in Air for 5 min.)

Alloy	No. Cycles	Mass Change mg/cm²
230	2337	-14.8
617	2337	-7.9

Oxygen Plus Sulfur Dioxide Test

The mass change results at 704°C (1300°F) for the oxygen plus sulfur dioxide test are shown in Figure 4. All alloys showed low rates of mass change in this test with the exception of alloys 214 and 230.

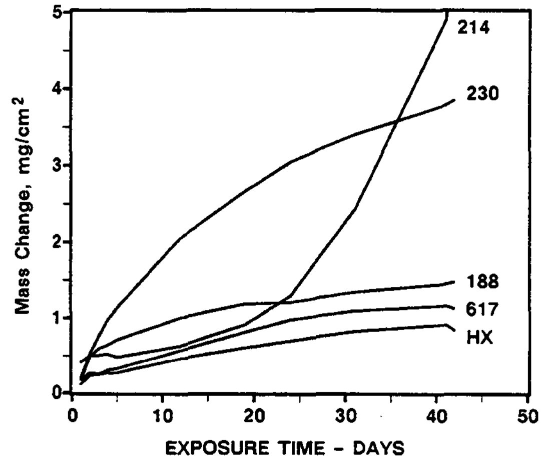

Figure 4. Mass Change Versus Exposure Time for Alloys Exposed at 704°C (1300°F) in Oxygen Plus 4% Sulfur Dioxide.

Carburization Resistance

It is known that the formation of a stable oxide scale on the surface improves the carburization resistance of an alloy (3-5). The presence of aluminum and a substantial amount of chromium in alloy 617 provides good resistance to carburization. The mass change data for various alloys in H_2-1%CH_4 and H_2-5.5%CH_4-4.5%CO_2 at 1000°C (1832°F) are shown in Figures 5 and 6 respectively. The mass changes for various alloys at the end of about 1000 hours in both H_2-1%CH_4 and H_2-5.5%CH_4-4.5%CO_2 are shown in Table 3. The data in Figure 5 and Table 3 indicate that the mass gain for alloy 617 is the least among all the alloys tested in H_2-1%CH_4. In particular the mass gains for alloys 188 and 230 are about 4 times that of alloy 617. Figure 7 shows microstructures of alloys 230 and 617 after an exposure of about 1000 hours in H_2-1%CH_4. The microstructures indicate more abundance of carbides both at the grain boundaries and within the grains for alloy 230 relative to alloy 617.

Table 3

Mass Change Data Per Unit of Exposed Surface Area for Alloys Exposed to Carburizing Environments at 1000°C for about 1000 h.

Environment→Alloy	H_2-1%CH_4 mg/cm^2	H_2-5.5%CH_4-4.5 %CO_2 mg/cm^2
309	61.8	-
800	45.6	31.9
625	38.7	14.7
DS	36.2	-1.1
330	35.1	7.3
HX	28.3	19.5
230	26.3	17.7
188	23.9	16.4
601	20.2	27.3
600	15.6	11.8
617	6.9	7.7

In the case of the H_2-5.5%CH_4-4.5%CO_2 environment, the mass gains for alloys 230 and 188 are about 2 times that for alloy 617. However, alloys 330 and DS showed lower mass gains than alloy 617 as shown in Figure 6 and Table 3. Both alloys 330 and DS contain about 1 to 2% Si which helps in reducing the carbon intake of the alloys by the formation of silica sub-scales in oxidizing carburization environments (6). Alloys 330 and DS are high temperature alloys used for industrial heat treatment applications (furnace parts, heat treatment baskets) that involve varying oxidizing and reducing conditions. Silicon is added to avoid internal oxidation and improve general oxidation and carburization resistance. However, addition of silicon reduces the creep strength and affects weldability. Therefore, silicon is not added to solid solution strengthened high nickel alloys where creep rupture and weldability are important. Alloy 617 has the best carburization resistance among all the high nickel alloys tested, with the exception of alloys 330 and DS, in the oxidizing carburization test.

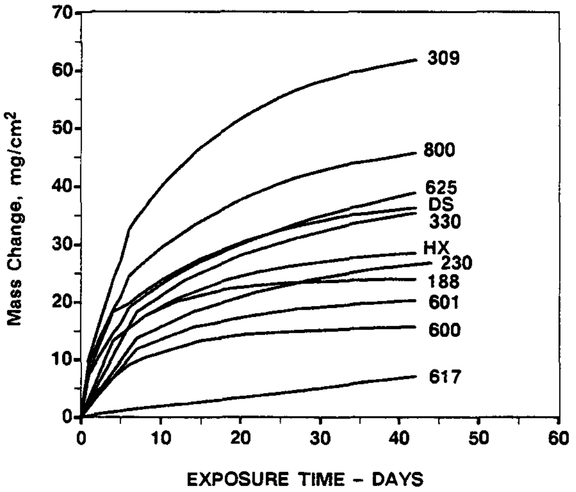

Figure 5. Mass Change Versus Exposure Time for Alloys Exposed to H_2-1%CH_4 at 1000°C for about 1000 h.

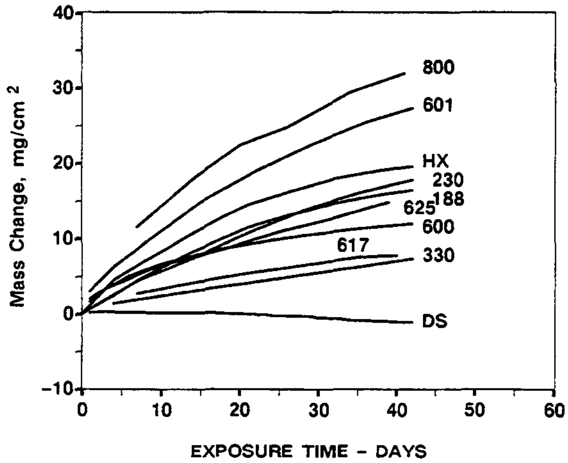

Figure 6. Mass Change Versus Exposure Time for Alloys Exposed to H_2-5.5%CH_4 -4.5%CO_2 at 1000°C for about 1000 h.

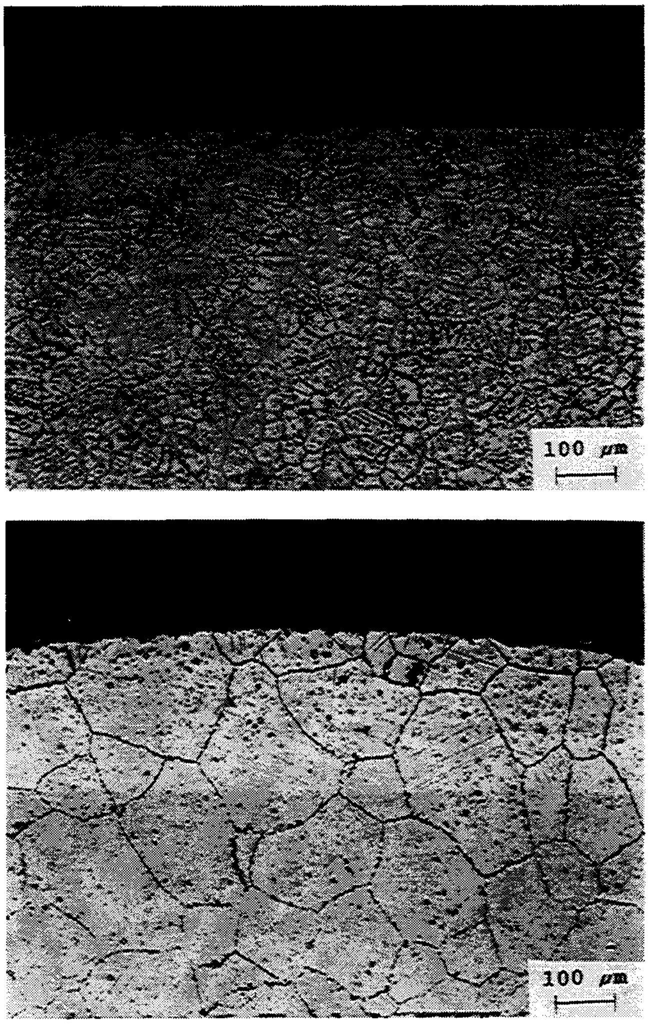

Figure 7. Optical micrographs obtained from cross-sections of samples of alloys 230 (top) and 617 (bottom) after exposure to H_2-1%CH_4 for 1008 hours at 1000°C. Both samples were etched in Murakami's reagent and micrographs taken at X100.

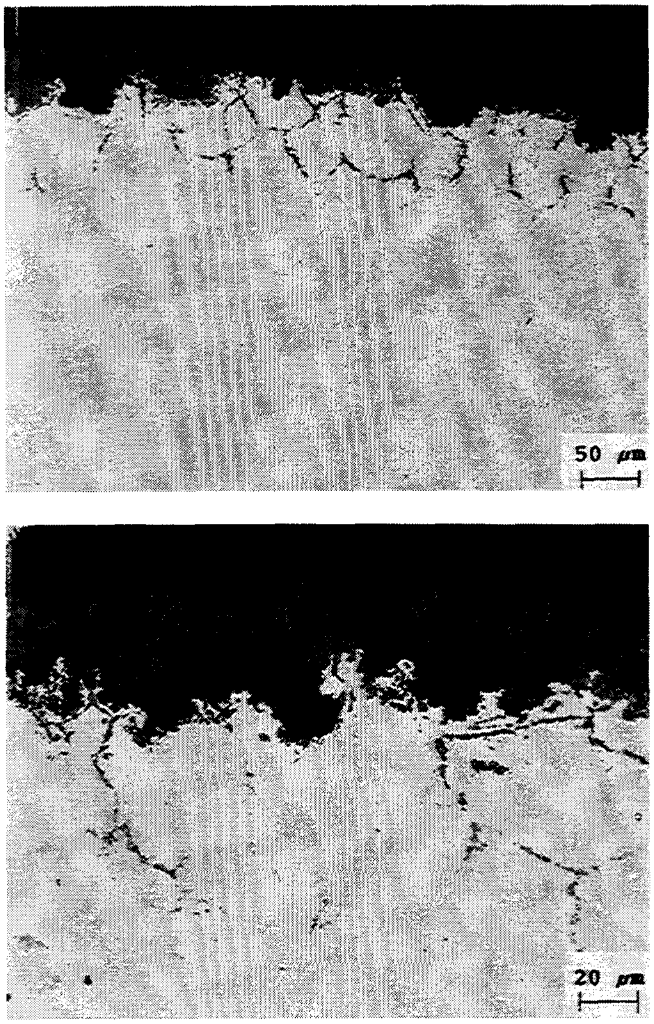

Figure 8. Micrographs of alloy 230 at 200X (top) and 500X (bottom) exposed to burner rig at 927°C for 500h. Note the grain boundary oxidation and necklace like chromium rich sulfides and pore formation at the scale metal interface in alloy 230.

Burner Rig Test

In the burner rig test mass loss data usually cannot be obtained during the test. However, the metal loss and depth of maximum attack are measured after exposure on mounted samples by metallography and the results are shown in Table 4. The results indicate that alloy 617 possesses excellent resistance to the oxidation/sulfidation environment found in the burner rig test. Typical optical micrographs obtained from alloys 230 and 617 after exposure to the burner rig environment at 927°C for 500 hours are shown in Figures 8 and 9. In alloy 230 substantial amounts of grain boundary oxidation and necklace like chromium rich sulfides can be noted. Also pore formation at the scale metal interface in alloy 230 is evident. It is these pores that make the scale weak and lead to scale spallation in alloy 230. In contrast, the scale is in general thinner on alloy 617 and is extremely adherent primarily due to aluminum rich pegs at the scale-metal interface.

Table 4

Metal Loss and Depth of Maximum Attack Data for Alloys in Burner-Rig Tests. The Air to Fuel Ratio Was Approximately 25:1, Using JP-5 Fuel (0.08%S).

Alloy	Time h	Temp. °C	Metal Loss Microns	Depth of Max. Attack Microns
230	500	927	46	163
617	500	927	25	53
HX	500	827	25	84
188	500	927	23	99
230	168	1066	716	968
617	312*	1066	602	744

* Note that the exposure time for alloy 617 is almost twice that of alloy 230 during this experiment.

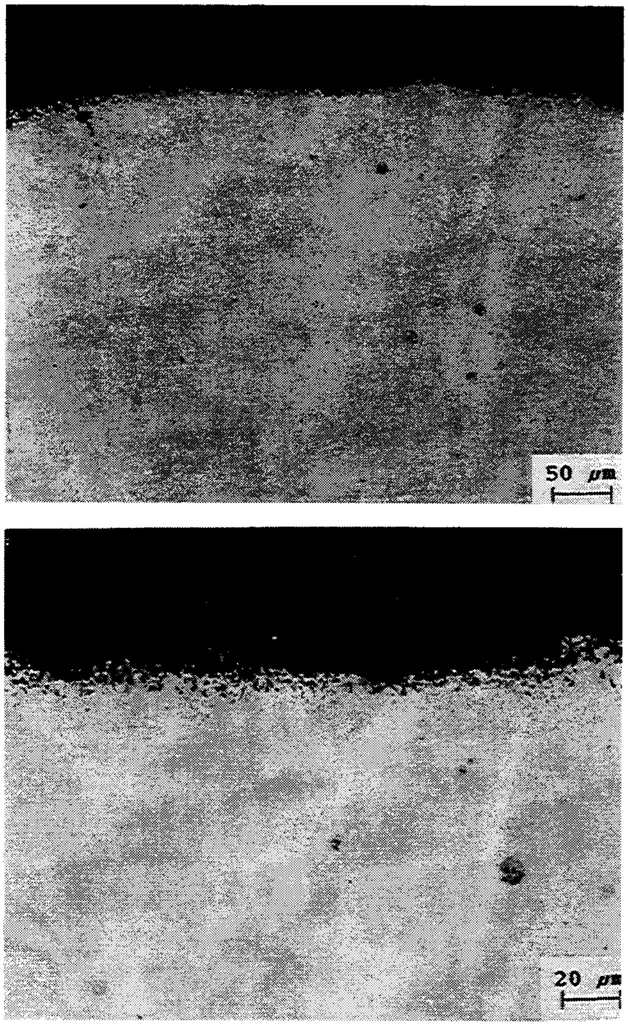

Figure 9. Micrographs of alloy 617 at 200X (top) and 500X (bottom) exposed to burner rig at 927°C for 500h. Note that the scale is in general thin and is extremely adherent primarily due to aluminum rich small pegs that can be seen at the scale-metal interface.

Optical micrographs from cross-sections of samples of alloys 230 and 617 after exposure to the burner rig at 1066°C are shown in Figures 10 and 11. On alloy 230, the scale has spalled off within 168 hours of exposure at this higher temperature due to pore formation at the scale metal interface similar to that shown in Figure 8. In the case of alloy 617, the scale is still intact even after 312 hours.

Both in cyclic oxidation and burner rig tests, alloy 617 showed better adhesion of scale to the base metal than alloy 230. The absence of void formation at the scale metal interface and the presence of oxide pegs protruding into the base metal are probably responsible for better scale adhesion in the case of alloy 617. Hou and Stringer (7) studied the effect of 1% Al on the oxidation behavior of a Ni-25% Cr alloy. These authors suggest that the irregular scale/metal interface with pegs of oxide extending into the base metal found in Ni-25Cr-1Al may be responsible in part for the improved adhesion of the scale to base metal. Similar explanation has been advanced by Giggins et al (8).

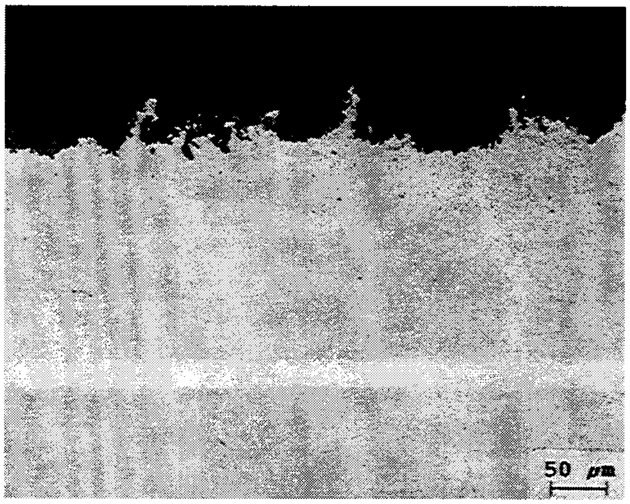

Figure 10. Micrograph of alloy 230 at 200X exposed to burner rig at 1066°C for 168h. Note that the scale has spalled off at this temperature due to pore formation at the scale metal interface similar to that shown in Figure 8.

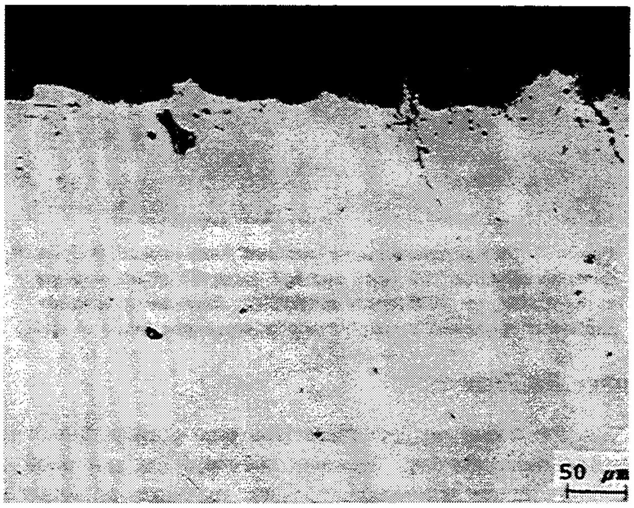

Figure 11. Micrograph of alloy 617 at 200X exposed to burner rig at 1066°C for 312h. Note that the scale developed on alloy 617 is still intact.

Allan et. al. (9) have shown the presence of interfacial voids at early times in the oxidation for poorly adherent scales. In this case, it has been suggested that the oxide particles act as a sink for vacancies generated during the oxidation which would otherwise condense at the interface forming voids (10). Oxide particles may also prevent impurities from segregating at the scale/metal interface. These impurities make the scale metal interface weak leading to poor adherence of scale to the base metal and causing the scale to spall earlier (11-12).

Impact Strength Measurements

Generally all these alloys have an impact strength of over 136 Joules (100 ft-lb) in the solution annealed condition. A substantial reduction in impact strength after prolonged exposure to intermediate temperatures is an indication that the alloy had undergone some microstructural change such as precipitation of carbides and intermetallic phases. The change in impact strength of various alloys after exposure to 649 to 982°C (1200 to 1800°F) for 100 and 500 hours is shown in Table 5. The results indicate that all the alloys except alloy 617 had substantial reduction in impact strengths after an exposure of only 100 hours at 760 to 871°C (1400 to 1600°F).

Table 5

Room Temperature Impact Test Results in Joules for Alloys after Exposure at the Indicated Temperatures for 100 and 500 h.

Alloy → Temperature	617	230	X	188	617	230	X	188
649°C (1200°F)	75	50	64	132	50	45	52	66
760°C (1400°F)	66	34	47	46	64	28	24	22
871°C (1600°F)	72	30	33	28	54	30	24	9
982°C (1800°F)	56	35	91	-	53	34	96	76

In an effort to identify the phases present after prolonged exposure, samples exposed at 871°C (1600°F) for 100 hours were dissolved electrolytically in 10% HCl- methanol solution and the resultant residues were analyzed by X-ray diffraction. The results of XRD analysis are shown in Table 6. The results clearly indicate the presence of an intermetallic phase (either mu or laves) at least for alloys HX and 188. In the case of alloy 230 the percentage of residue was much higher than other alloys. The weight of the residue could not account for carbides alone. XRD analysis indicated mainly carbides (carbonitrides?), titanium nitride and a small amount of another phase, possibly a form of mu phase, that could not be positively identified. Alloy 617 showed the least amount of residue and it was identified to contain only carbides. Earlier experiments conducted to study the effect of long term exposures (48000 hours) at 538°C to 760°C (1000°F to 1400°F) on the mechanical properties of alloy 617 indicate that alloy 617 retains good strength and tensile ductility (13). Additional impact strength data after long term exposure for alloy 617 is shown in Table 7 . These results conclusively show that alloy 617 does not form deleterious intermetallic phases after long term exposure to intermediate temperatures.

Table 6

XRD Analysis of Acid Extracted Residues of Impact Tested Samples after Exposure to 871°C (1600°F) for 100 h.

Alloy	Residue wt%	Phases Present
617	2.1	M_6C, $M_{23}C_6$
X	4.5	M_6C, $M_{12}C$, Mu
188	5.6	M_6C, $M_{23}C_6$, Laves
230	12.5	M_6C, $M_{23}C_6$, Mu?

Table 7

Impact Strength of INCONEL alloy 617 after Exposure to Elevated Temperatures

Exposure Temp. °C (°F)	Exposure Time h	Impact Strength Joules
No Exposure		171
595 (1100)	100	213
	1000	223
	4000	181
	8000	98
	12000	69
650 (1200)	100	191
	1000	35
	3640	35
	8000	40
	12000	38
705 (1300)	100	57
	1000	48
	4000	48
760 (1400)	100	56
	1000	63
	4000	62
	8000	64
	12000	67

CONCLUSIONS

Based on the results and discussion the following conclusions can be drawn about the performance of alloy 617:

1. In an oxidizing environment consisting of air + 5% water vapor at 1000°C alloy 617 is similar to alloy 230. However, alloy 617 is superior to alloy 230, 188 and HX in air plus 20% moisture.

2. In cyclic oxidation alloy 617 is superior to alloy 230, 188 and HX because of superior adhesion of the scale to the base metal.

3. Alloy 617 has excellent carburization resistance in H_2-1%CH_4 and H_2-5.5%CH_4-4.5%CO_2.

4. Alloy 617 has the best burner-rig oxidation/sulfidation resistance of the alloys tested.

5. Alloy 617 has good thermal stability at intermediate temperatures compared to alloys HX, 188 and 230.

ACKNOWLEDGEMENT

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