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**BURST TESTING OF ALLOYS 800 AND 310
AT 1255 K (1800°F) WITH A SIMULATED
COAL GASIFICATION ATMOSPHERE**

PREPARED BY AEROJET NUCLEAR COMPANY FOR

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

IDAHO OPERATIONS OFFICE UNDER CONTRACT E(10-1) -1375

MASTER

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**BURST TESTING OF ALLOYS 800 AND 310
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COAL GASIFICATION ATMOSPHERE**

by
C. E. Dixon

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ABSTRACT

Several corrosion- and heat-resistant alloys are being considered for long term applications in coal gasification plants at temperatures up to 1,255 K (1,800°F) in high pressure environments of mixed hydrogen, water, hydrocarbons, and sulfides. A method for *in situ* testing has been developed for short time mechanical tests of candidate alloys in high pressure, high temperature, gaseous environments, referred to as coal gasification atmosphere (CGA). The method involves bursting thin-walled tubes, using various gases to produce the burst hoop stress.

The short time 1,255 K (1,800°F) burst and creep rupture strength and ductility properties of Alloys 800 and 800H in a mixed gas environment, H₂, CO, CO₂, CH₄, SO₂ (CGA), are not reduced from properties obtained in air. However, the stress- and pressure-accelerated corrosion is more severe in CGA. It is expected that CGA will reduce long term strength and ductility in Alloy 800 as a result of the accelerated corrosion.

The short time 1,255 K (1,800°F) strengths of Alloy 310 in CGA and pure hydrogen environments are reduced from the values obtained in air by less than 10%. The ductilities (total circumferential elongation) are good -- approximately 20% for all test conditions. The CGA stress- and pressure-accelerated corrosion is greater than in air. Longer time tests in CGA are expected to result in additional strength degradation. Limited creep/fatigue tests of Alloy 310 in hydrogen show that hold times are significant. A greater cyclic life is observed using an 8-second hold time than a 55-second hold time.

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I. INTRODUCTION

Certain internal components of coal gasification pilot plants require exposure of metal parts to temperatures as high as 1,255 K (1,800°F)^[1]. Gas mixtures of H₂, CO₂, CO, N₂, H₂S, H₂O, and hydrocarbons at pressures up to 7.24 MPa (1,500 psi) are in contact with the alloys for long periods of time. Metallic materials for possible use as coal gasification plant internal components include Alloys 310, 50Cr-50Ni, 800, HK40, 309, 314, RA330. They may be used in either the coated or uncoated conditions. Designers are handicapped by lack of information on corrosion-erosion rates and effects of the gaseous environment on tensile, creep, and fatigue strengths.

It is well known that hydrogen gas can produce severe embrittling effects in many alloys over a wide range of pressures and temperatures^[2]. For example Chandler and Walter^[3] found many alloys to have severely reduced notched strength (and less severely reduced unnotched strength) based on short time tensile tests in the presence of clean high pressure hydrogen. The complete temperature dependence of this "hydrogen environment" embrittlement was not established; however, it was more severe at room temperature than at higher temperatures. Examples of these data from Reference 3 are shown in Tables I and II. Harris and VanWanderham^[4] observed the same phenomena as Chandler and Walter^[3] and in addition found reduced creep and fatigue resistance in some alloys when tested in hydrogen at 922 K (1,200°F).

McCoy^[5] investigated the effect of hydrogen on the creep resistance of several alloys. He found Nickel 200, Nickel 270, electron-beam zone-refined nickel, and high purity copper to have inferior creep resistance in hydrogen as compared to an argon atmosphere. On the other hand, Armco iron, electrolytic iron, and 304 stainless steel were not influenced by a hydrogen environment. The conditions for hydrogen embrittlement are very complex, and it is not safe to predict the presence or absence of the embrittlement for new test conditions. Many tests are required to establish reliable strength and ductility limits of materials for the new environments which will exist in current and proposed coal gasification plants.

The present experiments were undertaken to establish the value of a tube burst procedure for determining tensile and short term creep strengths of alloys in the presence of high pressure, high temperature hostile gases. The emphasis in these tests was to determine whether the short time strength and ductility properties of Alloys 800 and 310 were different as a result of the *in situ* gaseous environment. A gaseous environment can affect the properties by either bulk or surface effects. Bulk effects would be observed (a) if a gaseous constituent completely penetrates the alloy during the time of the test and consequently affects intra- or intergranular deformation or (b) if the presence of a gas at a crack tip can affect the crack growth rate. Surface effects would be observed (c) due to gross surface corrosion which would penetrate the surface uniformly and reduce the effective cross-sectional area of the alloy or (d) by nonuniform corrosion penetration which would result in surface cracks which eventually grow and fracture the specimen. Strength and ductility properties are compared for each alloy after bursting in air, a mixed gas (CGA),

TABLE I

MATERIALS EXTREMELY EMBRITTLED BY 10,000 PSI H₂ AT 72°F^[3]

Material	Strength Ratio (H ₂ /He)		Unnotched Ductility			
	Notched (K _t = 8.4)	Un- notched	Elongation (%)		Reduction of Area (%)	
			He	H ₂	He	H ₂
18 Ni-250 maraging	0.12	0.68	8.2	0.2	55	2.5
410 stainless steel	0.22	0.79	15	1.3	60	12
1042 Q&T	0.22	...	...	...	...	...
17-7 PH stainless steel	0.23	0.92	17	1.7	45	2.5
Fe-9Ni-4Co-0.20C	0.24	0.86	15	0.5	67	15
H-11	0.25	0.57	8.8	0	30	0
Rene 41	0.27	0.84	21	4.3	29	11
Electroformed Ni ^[a]	0.31	...	...	...	...	...
4140	0.40	0.96	14	2.6	48	9
Inconel 718	0.46	0.93	17	1.5	26	1
440 C	0.50	0.40	...	...	3.2	0

[a] Tested in 7000-psi H₂.

and high purity hydrogen. Considerable strength data are available for these alloys from tests in air at one atmosphere. It is urgent to know whether existing in-air strength data can be used in design or whether completely new strength data are required for the complex gaseous environments which are present in coal gasification systems.

TABLE II

MATERIALS SEVERELY EMBRITTLED BY 10,000 psi H₂ AT 72°F^[3]

Material	Notch Strength Ratio (H ₂ /H _e) (K _t = 8.4)	Unnotched Ductility			
		Elongation (%)		Reduction of Area (%)	
		He	H ₂	He	H ₂
Ti-6Al-4V (STA)	0.58	...	...	...	...
430 F	0.68	22	14	64	37
Nickel 270	0.70	56	52	89	67
A515	0.73	42	29	67	35
HY-100	0.73	20	18	76	63
A372 Class IV	0.74	20	10	53	18
1042 normalized	0.75	...	...	59	27
A533-B	0.78	...	...	66	33
Ti-6Al-4V (annealed)	0.79	...	...	...	...
AISI 1020	0.79	...	...	68	45
HY-80	0.80	...	...	70	60
Ti-5Al-2.5Sn ELI	0.81	...	...	45	39
Armco iron	0.86	...	...	83	50

II. CONCLUSIONS

1. MATERIAL PROPERTIES EFFECTS

1.1 Alloy 800 and 800H

The strength and ductility at 1,255 K (1,800°F) of Alloys 800 and 800H are not decreased by CGA from air values. Alloy 800H strength is not reduced by pure hydrogen. Alloy 800 CGA burst test specimens are penetrated to greater depths by corrosion than those burst with an air atmosphere. In longer time tests, it is expected that this CGA corrosion would degrade ductility and strength.

1.2 Alloy 310

The CGA and pure hydrogen environments may slightly decrease the 1,255 K (1,800°F) strength of Alloy 310 as compared with an air environment, but not more than 10%. Alloy 310 ductilities have a large scatter, but the ductility remains good (elongation is about 20%) in all environments.

2. TEST METHOD

Advantages:

- (1) The effects of a high pressure, high temperature, high stress environment on short time mechanical properties of materials can be determined with an inexpensive burst test apparatus.
- (2) Stress- or pressure-accelerated corrosion can readily be detected by this method.
- (3) Creep/fatigue testing can be performed by this method.
- (4) Short time creep rupture tests can be performed conveniently.

Disadvantages:

- (1) Long time mechanical tests by this method will be difficult to interpret since substantial corrosion may thin the tube walls nonuniformly.
- (2) Yield strength and uniform elongation could not be obtained in the present set up. It would be desirable to detect small plastic deformations with an external extensometer, an optical method, or a very sensitive pressure transducer.

III. MATERIALS AND ENVIRONMENTS

Alloys 800 and 310 were chosen for these preliminary tests. All tests were performed at 1,255 K (1,800°F). A mixture of 20% CO₂, 30% CO, 40% H₂, 10% CH₄, and 0.5% SO₂ was chosen as an approximate simulation of a coal gasifier atmosphere. This gas is abbreviated "CGA" in this report. Table III summarizes the material and gas chemistries. Extensive testing has been performed at Illinois Institute of Technology Research Institute (IITRI) under contracts with the Metal Properties Council and the U. S. Energy Research and Development Administration, using the above gases in the same proportions except that they were mixed with 40% H₂O, and H₂S was used rather than SO₂. The IITRI corrosion tests were performed primarily at gas pressures of 6.9 MPa (1,000 psi) and at 1,255 K (1,800°F).

Alloys 800 H and 310 were purchased from commercial stock as 1.59 cm (5/8 inch) diameter rod. These materials met the requirements of ASTM B408 and ASTM A276 specifications, respectively. Alloy 800 was obtained from a heat which had been previously studied for its low-cycle fatigue characteristics at 973 K (1,292°F) at the Idaho National Engineering Laboratory^[6].

"The ingot was hot-rolled at 1,503 K (2,246°F) with the final eight passes at 1,432 K (2,102°F) and given a final anneal at 1,313 K (1,904°F) and straightened. No other heat treatment was performed on the specimens prior to testing. Final hardness was HRB 75 with a grain size of ASTM 4-5 after aging".^[6]

This heat of Alloy 800 had a variety of grain sizes. A single check made on the stock used in the current tests showed a grain size of ASTM 7-10.

TABLE III

MATERIAL AND GAS CHEMISTRY

Alloy 310 ASTM A 276 Chemical Requirements										
<u>C</u> <u>max</u>	<u>Mn</u> <u>max</u>	<u>P</u> <u>max</u>	<u>S</u> <u>max</u>	<u>Si</u> <u>max</u>	<u>Cr</u>	<u>Ni</u>	<u>Fe</u>			
0.25	2.00	0.045	0.030	1.50	24.00-26.00	19.00-22.00	balance			
Alloy 310 Heat X 14099 Supplier's Certified Analysis										
<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Cr</u>	<u>Ni</u>	<u>Cu</u>	<u>Fe</u>		
0.07	1.63	0.025	0.016	0.56	25.23	19.90	0.29	balance		
Alloy 800 ASTM B 408 Chemical Requirements										
<u>C</u> <u>max</u>	<u>Mn</u> <u>max</u>	<u>Fe</u>	<u>S</u> <u>max</u>	<u>Si</u> <u>max</u>	<u>Cr</u>	<u>Ni</u>	<u>Cu</u> <u>max</u>	<u>Al</u>	<u>Ti</u>	
0.10	1.5	balance	0.015	1.00	19.0-23.0	30.0-35.0	0.75	0.15- 0.60	0.15- 0.60	
Alloy 800, Heat Nos. HH 7531A and HH 5633A (8COH)										
<u>Heat</u>	<u>C</u>	<u>Mn</u>	<u>Fe</u>	<u>S</u>	<u>Si</u>	<u>Cr</u>	<u>Ni</u>	<u>Cu</u>	<u>Al</u>	<u>Ti</u>
HH 7531A	0.03	1.05	46.48	0.007	0.26	19.25	32.44	0.16	0.14	0.20
HH 5633A	0.07	0.91	44.4	0.007	0.42	21.03	31.80	0.56	---	0.44
Simulated Coal Gasification Atmosphere (CGA)										
Supplier's Certified Analysis Cylinder 74192										
<u>CO₂</u>	<u>CO</u>	<u>H₂</u>	<u>CH₄</u>	<u>SO₂</u>	<u>O₂</u>					
20.0	29.9	39.6	10.0	0.49	---					
INEL Analysis										
<u>CO₂</u>	<u>CO</u>	<u>H₂</u>	<u>CH₄</u>	<u>SO₂</u>	<u>O₂</u>					
20.0	30.0	39.1	10.1	0.41	<0.05					

IV. TEST METHOD AND PROCEDURES

Conventional tensile and creep testing in a high temperature, high pressure, gaseous environment is difficult and expensive. The conventional tensile test procedures require a test environment chamber of one inch or more diameter which imposes a high hoop stress [for 1,255 K (1,800°F) and a reasonable wall thickness]. The environment chamber need not be used as the pressure vessel if a second vessel with cooler walls contains the environment chamber and an inert gas with pressure adjusted to keep the pressure drop across the high temperature environment chamber small. Such a system is complex and expensive. It is also difficult to introduce stressing fixtures into a high temperature pressure vessel.

The burst test which utilizes a small tubular specimen [0.952 cm (0.375 inch) inside diameter and 0.056 cm (0.022 inch) wall thickness in these tests] as the pressure vessel and applies stress by internal pressurization involves a much simpler system. The burst test has the disadvantage that the environmental gas pressure is not constant. The maximum pressure is determined by the specimen design and the strength of the material. The conditions used in the present experiment involved environmental gas pressures of about 6.2 to 10.3 Mpa (900 to 1,500 psi) to burst the two alloys. A limited number of short time creep and fatigue tests were conducted where the environmental gas pressures were in the range of 4.48 to 5.52 MPa (650 to 800 psi).

Good correlations are usually found between burst strength and tensile strength; thus the burst test is reliable for determining tensile strength. Correlations between uniaxial tensile ductility and circumferential ductility in a burst test are not well known, but generally the circumferential ductilities are much smaller than the uniaxial ductilities, because of the biaxial stress state. Ductility is usually not an explicit quantitative design requirement, other than the requirement that the percent elongation exceed a certain value to assure that a component can relieve high local stresses by a small amount of plastic flow.

A schematic of the burst test apparatus is shown in Figure 1. A photograph of the assembled apparatus is shown in Figure 2. Figure 3 shows a photograph of the pressurizing apparatus.

All specimens were fabricated to the requirements of Drawing 406408, shown in Figure 4. The burst strength was determined by noting the pressure at burst, and the maximum hoop strength was calculated using the thin wall formula, using the average mean diameter (mid-wall) and the average wall thickness. The dimensional variations of the specimens were small enough that all hoop stress calculations were based on the 0.056 cm (0.022 inch) wall thickness and 1.01 cm (0.397 inch) mean diameter. Thus the hoop stress was determined by the relation

$$\sigma = \frac{PD}{2t} = \frac{P \cdot 0.397}{2(0.022)} = 9.023 P$$

Fig. 1 Schematic of burst apparatus.

Fig. 2 Assembled burst apparatus.

Fig. 3 Screw-driven piston and cylinder pressurizer.

Fig. 4 Details of burst specimen. - Drawing 406408

where

- P = internal pressure
- D = mean diameter
- t = wall thickness.

End plugs, made of Alloy 347, were fusion-welded into the specimen. Prior to welding the specimens were almost filled with high density Al_2O_3 bars, 0.914 cm (0.360 inch) outside diameter, to minimize the volume of gas which escaped and exploded or burned in the furnace at the time of the burst.

A 0.318 cm (1/8 inch) OD, 0.14 cm (0.055 inch) ID, Alloy 316 capillary tube was fusion-welded into one end plug and connected to the pressurizing system by pressure fittings. Prior to welding, all specimens were ultrasonically cleaned in acetone and welded using clean white glove handling. Chromel-Alumel thermocouples [0.03 cm (12 mil) diameter] were attached at the radius on each end of the specimen. The specimen was inserted into the furnace when the furnace temperature was slightly below 1,255 K (1,800°F). The capillary tube was attached to the pressurizing system within about 30 seconds and pumped down with a mechanical vacuum pump. When the temperature reached equilibrium at $1,255 \pm 3$ K ($1,800 \pm 5^\circ\text{F}$), usually within less than 10 minutes, the vacuum system was valved off, and the pressurizing system was prepressurized from a gas bottle to pressures of 2.07 MPa (300 psi) for the Alloy 800 tests and 4.14 MPa (600 psi) for the Alloy 310 tests. The pressurizing piston was then actuated; it traveled at a rate of 0.203 cm (0.080 inch)/min. The pressure increased nonlinearly with time until the specimen burst or leaked. The burst was detected from a rapid pressure drop. The highest pressure was used to calculate the burst strength. The pressurization rate was nonlinear due to the constant rate of movement of the piston and the compressibility of the gas. The magnitude of the strain rate sensitivity of the burst strength was not realized during the Alloy 800 and Alloy 310 tests, and, in some instances there is some ambiguity in determining whether burst strength values are characteristic of the environment or the strain rate. The test data in Tables IV, V and VI show the pressurizing rate at fracture in order to help resolve any ambiguities about the reason for strength change. The strain rate while the specimen was undergoing plastic deformation was about 0.05/minute. The strain rate in the elastic region was much lower. The time required to pressurize and burst the specimens was typically 20 minutes.

TABLE IV

ALLOY 800

1,255 K (1,800°F) Burst Strengths and Total Circumferential Elongation (TCE)

Specimen Number	Burst Gas	Burst Stress		TCE (%)	Burst Pressure		Pressurizing Rate		Type of Failure
		(MPa)	(ksi)		(MPa)	(psi)	(kPa)	(psi/min)	
11-1	air	63.4	9.2	23	7.0	1,017	379	55	rupture
3-1	air	58.6	8.5	30	6.5	945	290	42	rupture
Average		61.4	8.9	26	6.9	1,002	330	48	
7-1	CGA ^[a]	60.0	8.7	22	6.7	965	496	72	rupture
8-1	CGA	62.0	9.0	31	6.9	998	552	80	rupture
9-1	CGA	57.9	8.4	21	6.4	933	469	68	pinhole
Average		60.0	8.7	25	6.7	965	505	73	
4-1	H ₂	54.5	7.9	26	6.0	875	165	24	pinhole

1255 K (1800°F) Rupture Tests at 40.4 MPa (5865 psi) Hoop Stress

Specimen Number	Burst Gas	Constant Hoop Stress		TCE (%)	Time to Rupture	Type of Failure
		(MPa)	(psi)			
13-1	air	40.4	5865	10	20 min.	fissure
14-1	air	40.4	5865	12	20 min.	pinhole
2-1	CGA	40.4	5865	11	12 min.	fissure
6-1	CGA	40.4	5865	18	26 min.	fissure

[a] Nominal composition: 20% CO₂, 30% CO, 40% H₂, 10% CH₄, 0.5% SO₂.

Four short time creep rupture tests were performed with Alloy 800. The test procedure was the same as the burst procedure except that the pressurizing piston was stopped when the pressure had reached 4.5 MPa (650 psi) [40.4 MPa (5.9 ksi) hoop stress]. The pressure was maintained constant until the specimen burst or leaked.

The outside surface, OD, of all Alloy 800 specimens was exposed to air at about 1,255 K (1,800°F) while waiting for the specimen temperature to reach equilibrium and during the time of the test. A typical time to reach equilibrium was 10 minutes; thus the total time in which the OD was subject to 1,255 K (1,800°F) air corrosion was about 30 minutes. Air corrosion layers observed metallographically on the OD were less than 0.0025 cm (1 mil) thick.

TABLE V

ALLOY 800H

(Heat HH 5633A) 1,255 K (1,800°F) Burst Strength and Total Circumferential Elongation (TCE)

Specimen Number	Burst Gas	Burst Stress		TCE (%)	Burst Pressure		Pressurizing Rate		Type of Failure
		(MPa)	(ksi)		(MPa)	(psi)	(kPa)	(psi)/min	
22-1	air	84.8	12.3	21	9.4	1362	738	107	pinhole
28-1	air	87.6	12.7	21	9.7	1410	800	116	rupture
Average		86.2	12.5	21	9.6	1386	772	112	
20-1	air	75.8	11.0	19	8.4	1220	427	62	pinhole
25-1	air	73.8	10.7	14	8.2	1185	414	60	pinhole
Average		75.1	10.9	16	8.3	1202	421	61	
23-1	H ₂	77.9	11.3	21	8.7	1255	441	64	pinhole
21-1	H ₂	79.3	11.5	14	8.8	1280	427	62	pinhole
Average		78.6	11.4	18	8.7	1268	434	63	
24-1	CGA	73.8	10.7	35	8.1	1180	386	56	fissure
34-1	CGA	75.2	10.9	16	8.3	1205	400	58	pinhole
Average		74.5	10.8	26	8.2	1192	393	57	
27-1	argon	70.3	10.2	23	7.8	1125	331	48	pinhole
29-1	argon	74.5	10.8	27	8.3	1205	400	58	pinhole
Average		72.4	10.5	25	8.0	1165	365	53	

The test procedures for Alloy 310 and Alloy 800H were modified to minimize the extent of OD air corrosion since it appeared that some of the Alloy 800 fractures were initiated at OD surface cracks. The Alloy 310 and 800H specimens were encapsulated with an Alloy 304 tube which was fusion-welded in argon at each shoulder of the specimen, leaving a closed OD annulus between specimen and tube.

When the closed OD annulus containing argon was at 1,255 K (1,800°F), the pressure increase due to the temperature decreased the pressure differential across the specimen wall. A 207 kPa (30 psi) correction to the internal burst pressure was made for this reason when the annulus was not vented. In most specimen assemblies the OD annulus was vented because it was discovered that the hydrogen diffusion rate through the specimen wall at 1,255 K (1,800°F) was sufficient to increase the gas pressure in the OD annulus. In one case using pure hydrogen for the burst gas, a test lasted 20 minutes, and the hydrogen pressure in

TABLE VI

ALLOY 310

1,255 K (1,800°F) Burst Strength and Total
Circumferential Elongation (TCE)

Specimen Number	Burst Gas	Burst Stress		TCE (%)	Burst Pressure		Pressurizing Rate		Type of Failure
		(MPa)	(ksi)		(MPa)	(psi)	(kPa)	(psi)/min	
2-2	air	93.8	13.6	25	10.4	1505	1379	200	rupture
9-2	air	90.3	13.1	15	10.0	1453	1241	180	rupture
Ratio	1.00	91.7	13.3	20	10.2	1479	1310	190	
40-2	H ₂	82.0	11.9	20	9.1	1320	855	124	rupture
43-2	H ₂	88.9	12.9	23	9.9	1430	1048	152	rupture
Ratio	0.93	85.5	12.4	22	9.5	1375	951	138	
8-2	H ₂	75.8	11.0	35	8.4	1215	372	54	pinhole
10-2	H ₂	74.5	10.8	21	8.2	1192	372	54	pinhole
Ratio	0.81	75.2	10.9	28	8.3	1204	372	54	
1-2	CGA	82.0	11.9	23	9.1	1320	965	140	rupture
16-2	CGA	85.5	12.4	16	9.5	1380	1158	168	fissure
13-2	CGA	84.8	12.3	22	9.4	1360	462	67	fissure
Ratio	0.92	84.1	12.2	20	9.3	1353	855	124	
6-2	argon	77.2	11.2	25	8.6	1245	434	63	pinhole
7-2	argon	86.9	12.5	10	9.6	1390	552	80	pinhole
11-2	argon	85.5	12.4	17	9.5	1376	290	42	fissure
Ratio	0.90	83.4	12.1	17	9.2	1337	421	61	

Alloy 310 Creep-Fatigue Tests

Specimen Number	Burst Gas	Max Hoop Stress		TCE (%)	Cycles to Failure
		(MPa)	(ksi)		
5-2 [a]	air	49.6	7.2	10	68
4-2 [a]	air	49.6	7.2	20	116
14-2 [a]	H ₂	49.6	7.2	14	81
17-2 [a]	H ₂	49.0	7.1	—	251

[a] Pressurize from bottle to 5.5 MPa (800 psi) [49.6 MPa (7218 psi) hoop stress] hold for 1 min., vent gas, and repeat until burst or leak.

[b] Same as above except total cycle time was 10 sec.

the OD annulus was estimated to be 3.4 MPa (500 psi). In subsequent hydrogen burst tests the OD annulus was vented to prevent the build up of hydrogen pressure. The Alloy 310 CGA burst test used both the unvented and vented OD annulus. Since the apparent burst strengths were the same in both vented and unvented capsules, it was assumed that a significant amount of hydrogen had not diffused through the Alloy 310 wall in the time of the test.

Four creep/fatigue tests were performed at 1,255 K (1,800°F) with Alloy 310. The specimens were pressurized rapidly to 5.5 MPa (800 psi) [49.6 MPa (7.2 ksi) hoop stress] directly from a gas bottle; pressure was held for about one minute. The specimen was vented rapidly and immediately repressurized to 5.5 MPa (800 psi) and held for about one minute. This procedure was repeated until the specimen burst or leaked. In a typical cycle the depressurizing and repressurizing time was about five seconds; the pressure was held sufficiently long to make the total cycle 60 seconds in length.

A single specimen was tested in the same manner except that a total cycle time of 10 seconds was used. In this test the depressurization-repressurization time was about two seconds; thus, the average hold time was eight seconds. This specimen had a cyclic life about three times that of one using a 55-second hold time; therefore, the hold time is significant in determining the cyclic life of a material.

V. TEST RESULTS

Alloy 800 (Heat HH 7531A) strength data using different gas environments are shown in Table IV. No significant differences in strength or ductility were observed between specimens which were burst with air or CGA. A single specimen was burst with hydrogen. This specimen had a 10% lower strength than those using air or CGA. Since the pressurization rate for the hydrogen test was 1/2 to 1/3 of that used in the other environments, the low value is attributed to the low strain rate. In this specimen the fracture appeared to originate on the inside surface, contrary to the case of the air and CGA bursts where the fracture originated at the outside surface. No ductility differences were detected as a result of using different gas environments.

The stress-rupture data in Table IV show considerable data scatter. From these data it cannot be determined if the small differences in rupture life may be a result of environmental effects. The ductilities at fracture in the stress-rupture tests were about half of those observed in the burst tests.

Alloy 800H (Heat HH5633A) strength data using the different gas environments are shown in Table V. In these tests the pressurization rate was held nearly constant for all tests except two in air, in which the rate was deliberately doubled. The effect of increasing the strain rate from 61 to 112 psi/min increased the average burst strength in air by 15%. The ratios of the average strength in the different environments to the strength in air, 74.4 MPa (10.8 ksi) at 420 kPa (61 psi)/min) are: hydrogen-1.05, CGA-0.99, argon-0.97. These differences are not significant for the sample size. No metallographic studies were made of Alloy 800H because of its presumed similarity to Alloy 800.

Alloy 310 strength and ductility data are shown in Table VI. The data scatter for a given condition is reasonable for this kind of test. The largest spread between minimum and maximum values was 8% for two data groups, whereas, the spread for the other three data groups was less than 5%.

Average values from Table VI and comparisons with the air burst strength are as follows:

Burst Gas	Average Burst Stress		Ratio to Burst Stress in Air	Average Pressurization Rate	
	MPa	ksi		MPa	(psi)/min
Air	91.6	13.3	1.00	1.31	190
H ₂	85.5	12.4	0.93	0.95	138
H ₂	75.1	10.9	0.81	0.37	54
CGA	84.1	12.2	0.92	0.85	124
Argon	83.4	12.1	0.90	0.42	61

The effect of strain rate (pressurization rate) on strength is not accurately known. However, it is well established that tensile strengths increase with increasing strain rates^[7].

VI. DISCUSSION

1. ALLOY 800 AND 800H STRENGTH AND DUCTILITY AT 1,255 K (1,800°F)

The average burst strength at 1,255 K (1,800°F) of Alloy 800 in air is 61.3 MPa (8.9 ksi). This can be compared with literature data in which a typical uniaxial strength is 68.9 MPa (10 ksi)^[8]. The total circumferential elongation averages 26%, which is much lower than literature values for uniaxial tensile elongation of about 115%^[8]. On the basis of both theory and experience, Weiss^[9] concludes that fracture ductility resulting from biaxial stresses is often reduced by as much as a factor of five as compared to uniaxial fractures. Hence the 26% value is reasonable. There is no significant difference in burst strength between the air and the CGA environments.

Only a single specimen was burst in hydrogen. The strength was about 10% lower than the air and CGA burst specimens. This difference is attributed to the low strain rate used in the hydrogen test rather than to an environmental effect.

Alloy 800H (Heat HH5633A) has a higher carbon content than Alloy 800. Alloy 800 has 0.03% C, whereas Alloy 800H contains 0.07% C. At approximately the same pressurization rate, 365-434 kPa (53-63 psi)/min, the strength of 800H is not significantly affected by any environment. Therefore, the environments hydrogen, CGA, and argon do not degrade the strength of 800H from the values measured in an air environment.

2. ALLOY 800 (HH 7531A) CREEP RUPTURE TESTS AT 1,255 K (1,800°F)

A hoop stress of 40.6 MPa (5.9 ksi) was chosen for the short time creep tests with the intention of having rupture times of about three hours. If the rupture life was influenced by grain boundary corrosion, three hours should be sufficient time to observe a degradation. The choice of stress was too high and the resulting corrosion times were comparable to the test times required for the burst test (about 20 minutes). Additional creep testing at lower stress levels is desirable so that effects on the rupture life of corrosion by the different gases can be determined.

3. ALLOY 800 METALLOGRAPHY

Metallographic studies of Alloy 800 are shown in Figures 5 through 18. The micrographs in Figure 6 and Figure 8, after gas bursts using air and CGA, respectively, suggest the fracture was initiated at cracks on the outside surface. This may explain why the

SPECIMEN 11-AIR

(A)

(B)

(C)

(D)

Fig. 5 Alloy 800, specimen 11 after 1,255 K (1,800°F) burst by air: (A) 1X, (B) 3X, (C) 5X transverse section, (D) 10X, inside diameter showing fracture surface. (Reduced in reproduction.)

SPECIMEN 11-AIR
Transverse Section at 200X

Fig. 6 Micrograph of Alloy 800, transverse section at 200X, after 1,255 K (1,800°F) burst by air: (A) and (B) fracture surfaces, (C) near fracture, (D) 180° from fracture. Note the uniform corrosion layer at the inside diameter. Outside diameter has thinner corrosion layer and small cracks [see (D)]. (Reduced in reproduction.)

SPECIMEN 7-CGA

(A)

(B)

(C)

(D)

Fig. 7 Alloy 800, after 1,255 K (1,800°F) burst by CGA: (A) 1X, (B) 3X, (C) 5X transverse section, (D) 10X inside diameter. Note the numerous cracks parallel to the fracture surface in comparison with the few after air burst in Figure 5 (D). (Reduced in reproduction.)

SPECIMEN 7-CGA
Transverse section at 200X

Fig. 8 Micrograph of Alloy 800, transverse section at 200X after 1,255 K (1,800°F) burst by CGA: (A) and (B) fracture surfaces, (C) near fracture, (D) 180° from fracture. Note the blunted cracks at inside diameter and greater grain boundary corrosion penetration than observed after the air burst. Outside diameter has many small cracks along grain boundaries, [see (D)]. (Reduced in reproduction.)

SPECIMEN 4-H₂

(A)

(B)

(C)

Fig. 9 Alloy 800 after 1,255 K (1,800°F) burst by hydrogen: (A) 1X, (B) 6X shows multiple cracks and pinholes, (C) 5X transverse section. (Reduced in reproduction.)

SPECIMEN 4-H₂
Transverse Section at 200X

Fig. 10 Micrograph of Alloy 800, transverse section at 200X, after 1,255 K (1,800°F) burst by hydrogen: (A) and (B) fracture area, (C) adjacent to fracture. Corrosion layer is very thin on both surfaces. Surface grain boundary cracks are present on both inside and outside surfaces. (Reduced in reproduction.)

(A)
(800)
6-1
CGA
CREEP

(B)
(800)
13-1
AIR
CREEP

Fig. 11 Alloy 800:(A) $\approx$ 5X of outside surface after 1,255 K (1,800°F) creep burst by CGA at hoop stress of 40.4 MPa (5,865 psi), shows multiple cracks near pinhole break; (B) $\approx$ 5X of outside surface after 1,255 K (1,800°F) creep burst by air. Circumferential crack is not typical. (Reduced in reproduction.)

(800)
6-1
200X
RUPTURE
CGA
CREEP

ID

Fig. 12 Micrograph of Alloy 800, 200X of transverse section at fracture after 1,255 K (1,800°F) creep burst by CGA at hoop stress of 40.4 MPa (5,865 psi); shows intergranular fracture. Rupture time was 12 min. (Reduced in reproduction.)

(800)
13-1
200X
RUPTURE
AIR
CREEP

ID

Fig. 13 Micrograph of Alloy 800, 200X of transverse section at fracture after 1,255 K (1,800°F) creep burst by air at hoop stress of 40.4 MPa (5,865 psi). Rupture time was 20 min. (Reduced in reproduction.)

(800)
6-1
200X
ADJACENT
TO
RUPTURE
CGA
CREEP

Fig. 14 Micrograph of Alloy 800, 200X of transverse section near fracture area after 1,255 K (1,800°F) creep burst by CGA at hoop stress of 40.4 MPa (5,865 psi); shows intergranular fracture. (Reduced in reproduction.)

(800)
13-1
200X
ADJACENT
TO
RUPTURE
AIR
CREEP

Fig. 15 Micrograph of Alloy 800, 200X of transverse section near fracture after 1,255 K (1,800°F) creep burst by air at hoop stress of 40.4 MPa (5,865 psi). (Reduced in reproduction.)

(800)
6-1
200X
180° FROM
RUPTURE
CGA
CREEP

Fig. 16 Micrograph of Alloy 800, 200X of transverse section, 180° from fracture, after 1,255 K (1,800°F) creep burst by CGA at hoop stress of 40.4 MPa (5,865 psi). Note the large amount of penetration by corrosion as compared with air rupture specimen in Figure 17. (Reduced in reproduction.)

(800)
13-1
200X
180° FROM
RUPTURE
AIR
CREEP

Fig. 17 Micrograph of Alloy 800, 200X of transverse section, 180° from fracture after 1,255 K (1,800°F) creep burst by air at hoop stress of 40.4 MPa (5,865 psi). Rupture time was 20 min. (Reduced in reproduction.)

(A)
(310)
2-2
200X
AT
FRACTURE
AIR

↙ ID

(B)
(310)
2-2
200X
180° FROM
FRACTURE
AIR

↙ ID

Fig. 18 Micrograph of Alloy 310, transverse section at 200X after 1,255 K (1,800°F) burst by air: (A) fracture area, (B) 180° from fracture. Note the multiple cracks at both surfaces.

burst strength was the same for each gas. The weakest part of the specimen was probably the outside surface, which was subject to corrosion by the air during the test. It may be significant that the deepest cracks on the inner surface of the CGA specimen were blunted (Figure 8); therefore, they are less subject to rapid crack propagation. Whatever the reason, it can be concluded that the short time strength properties of Alloy 800 are not degraded by CGA exposure. In comparing Figure 8 with Figure 6, the grain boundary corrosion at the inside surface of the CGA specimen (Figure 8) is seen to be much deeper than the air burst specimen. It seems likely that this grain boundary penetration would weaken the material during a test which lasted several hours.

The hydrogen burst specimen is shown in Figures 9 and 10. The fracture in Figure 9(B) shows multiple pinhole cracks rather than the larger ruptures characteristic of the air and CGA bursts. This mode of fracture is believed to be due to the low strain rate^[7]. The outside surface was a light gray, a definite indication of the presence of a reducing atmosphere during testing. It is concluded that the hydrogen diffusion through the 0.056 cm (0.022 inch) wall was sufficient to reduce oxidation by the external air during the test. Conclusive evidence of rapid diffusion of hydrogen through the 0.056 cm (0.022 inch) wall of Alloy 310 was obtained during a later test. The fracture area shown in Figure 10 suggests that the fracture is initiated at the interior surface of the hydrogen burst specimen, unlike the case for the air and CGA bursts. The scanning electron microscope (SEM) fractography, which is discussed in Appendix A, shows prominent grain boundary cracks unlike the air and CGA bursts.

4. ALLOY 310 STRENGTH AND DUCTILITY AT 1,255 K (1,800°F)

The average 1,255 K (1,800°F) burst strength in air at a strain rate of about 0.04/min is 74.4 MPa (10.8 ksi). This compares with literature values for tensile properties which range from 68.9 to 103 MPa (10 to 15 ksi) at 1,255 K (1,800°F). It is presumed that the tensile data were obtained at the strain rate 0.05 ± 0.01 /min recommended in ASTM E21. Thus the burst strength in these tests is comparable to those obtained by uniaxial tensile tests. Elongation data in the literature ranges between 25 and 55% at 1,255 K (1,800°F).

Alloy 310, unlike Alloy 800, shows a slight degradation due to hydrogen, CGA, and argon. It is believed that low strain rates, 3.72 kPa (54 psi)/min in hydrogen and 4.27 kPa (62 psi/min) in argon, contribute to the degradation. Although the issue of environmental effects may be obscured by variations in strain rate, it is believed that an upper limit on the environmental strength degradation in these short time tests is 10%. At the onset of these tests it was feared that strength degradation due to hydrogen gas mixtures might be much larger than 10%.

5. ALLOY 310 CREEP/FATIGUE AT 1,255 K (1,800°F)

The three exploratory creep/fatigue tests with a 55-second hold time at the maximum stress could not distinguish between air and hydrogen environments. Since low-cycle fatigue life is usually more dependent on short time ductility than on strength, this is not surprising. The single fatigue test with an 8-second hold time at the maximum stress showed increased cyclic life by a factor of about three over the other specimens with the 55-second hold time; thus the cyclic life of Alloy 310 appears to be sensitive to creep hold times even at times less than one minute.

6. ALLOY 310 METALLOGRAPHY

The "as-received" microstructure of Alloy 310 had a range of grain sizes between ASTM 3 and ASTM 7. Good comparisons of "as-received" and "after-fracture" grain sizes could not be made. The etching characteristics of both Alloy 800 and Alloy 310 were different "as-received" from "after-fracture". In the latter condition grain boundaries were not clearly revealed.

All specimens "after-fracture" in Figures 18-20 show numerous small surface cracks on both the inner and outer surfaces. The origin of fracture could not be determined. The mode of fracture was mixed intra- and intergranular.

An unusual corrosion penetration can be observed in Figure 19 for the CGA burst specimen. Individual grains were penetrated along preferred crystallographic planes. These planes are probably slip planes, and the corrosion rate is enhanced by the concentrated dislocation distributions. A comparison of the Alloy 800 corrosion by CGA in Figure 8 with the corrosion of Alloy 310 by CGA in Figure 19 indicates Alloy 310 to be more resistant to CGA corrosion than Alloy 800.

Fig. 19 Micrograph of Alloy 310, transverse section at 200X after 1,255 K (1,800°F) burst by CGA: (A) fracture area, (B) 180° from fracture. Note the corrosion penetration of grains at the inside surface along certain crystallographic directions.

(A)
(310)
10-2
200X
AT
FRACTURE
H₂
BURST

(B)
(310)
10-2
200X
NEAR
FRACTURE
H₂
BURST

Fig. 20 Micrograph of Alloy 310, transverse section at 200X after 1,255 K (1,800°F) burst by hydrogen: (A) fracture area, (B) near fracture area.

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APPENDIX A

SCANNING ELECTRON MICROSCOPE (SEM) FRACTOGRAPHY

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APPENDIX A

SCANNING ELECTRON MICROSCOPE (SEM) FRACTOGRAPHY

75 - ALLOY 310 BURST BY HYDROGEN, SPECIMEN 8-2

- 75 C1 10X Main longitudinal crack on inner surface. Fracture almost obliterated on OD - good crack on ID. Shows main crack and several smaller cracks - all axial, with irregular shape.
- 75 C2 100X Portion of main crack and a secondary crack. Appear to be intergranular and general.
- 75 C3 500X Portion of main crack - same as part of 75 C2. Crack appears to be predominantly intergranular. Virtually every grain appears to be cracked all around - individual grains very distinct.
- 75 C4 500X General view of ID showing cracking around each grain.
- 75 C5 2500X Small portion of crack from 75 C3. Magnification so high that perspective of overall sample is lost, but individual grains are even more evident.

76 - ALLOY 310 BURST BY CGA, SPECIMEN 13-2, INNER SURFACE

- 76 C1 10X Sample ID at failure region. View in this SEM photomicrograph shows the sample tilted 40° to reveal the ID. One major crack and many secondary cracks are visible. Cracks are generally axial but somewhat irregular in shape. Some circumferential features can also be seen. Several rather prominent; may be machining marks. Others are quite fine and very numerous.
- 76 C2 100X Portion of main crack on sample ID. Close-up of the bottom portion of the main crack in 76 C1. Appears to be many, very small secondary cracks near the main crack. Individual grains cannot be identified; hence it is not possible to tell if the main crack is transgranular or intergranular. The fine, circumferential striations are seen to cover the entire sample in this view.
- 76 C3 500X Portion of main crack on sample ID. Higher magnification of a part of the crack seen in 76 C2. Secondary cracks seem to follow along grain boundaries. Individual grains are still not totally defined. The main crack, at least in this area, is following some grain boundaries, but the fracture doesn't really have a classical "intergranular" appearance. In this view, there appear to be surface deposits.
- 76 C4 2500X Portion of main crack on sample ID. Higher magnification of part of the crack seen in 74 C3. This part of crack does appear more intergranular. Surface is covered with tiny particles which may be corrosion product from the CGA atmosphere.

77 - ALLOY 310 BURST BY CGA, SPECIMEN 13-2, OUTER SURFACE

- 77 C1 10X Sample OD at failure region. This view is similar to 76 C1, showing one major crack and many smaller cracks. All are either axially oriented or appear as pinholes. Crack shape is irregular.
- 77 C2 100X Portion of main crack on sample OD. Fracture mode hard to identify, but it does look like it may be more ductile than that seen on the ID. A few circumferential striations visible that may be machining marks.
- 77 C3 500X Portion of main crack on sample OD. Higher magnification view of an area from 77 C2. Fracture mode does appear fairly ductile in this view. Also, many small, secondary cracks are visible. Individual grains cannot be distinguished.
- 77 C4 2500X Portion of main crack on sample OD.

78 - ALLOY 310 BURST BY AIR, SPECIMEN 2-2, FRACTURE SURFACE

- 78 C1 11X Fracture surface. The specimen split open in a long, axial break. There is a laminar appearance in the fracture.
- 78 C2 110X Fracture surface. A fairly typical appearance for a ductile type of failure.
- 78 C3 550X Fracture surface. Part of the area shown in 78 C2. Looks more distinctly like ductile dimples. Discrete particles are in some of the holes, which may be either non-metallic inclusions which were present in the "as-received" material or corrosion products.
- 78 C4 2750X Fracture surface. High magnification of an area from 78 C3. Inclusions are shown more distinctly. One inclusion appears to have fractured.

79 - ALLOY 310 BURST BY AIR, SPECIMEN 2-2, ADJACENT TO FRACTURE ON INNER SURFACE

- 79 C1 100X Sample ID adjacent to fracture. This view of the ID shows many secondary cracks running parallel to the failure. They are slightly irregular in shape but generally straight and axial. Individual grains cannot be distinguished, but, from their length and shape, it seems that many of the cracks go across grains in a ductile manner rather than by cleavage. This view also shows the circumferential marks that were seen on sample 13-2 - probably from machining.
- 79 C2 500X Sample ID adjacent to fracture. Higher magnification of an area from 79 C1. These are definitely not cleavage cracks, and they don't appear to be entirely intergranular; so they are probably ductile across the grains.

79 C3 2500X Sample adjacent to fracture. Higher magnification of an area from 79 C2.
There appears to be a surface corrosion product present, rather similar to 76 C4.

75 C1
8-2
10X
ID
H₂

75 C2
8-2
100X
ID
H₂

75 C3
8-2
500X
ID
H₂

75 C4
8-2
500X
ID
H₂

75 C5
8-2
2500X
ID
H₂

76 C1
13-2
100X
ID
CGA

76 C2
13-2
100X
ID
CGA

76 C3
13-2
2500X
ID
CGA

76 C4
13-2
2500X
ID
CGA

77 C1
13-2
10X
OD
CGA

77 C2
13-2
100X
OD
CGA

77 C3
13-2
500X
OD
CGA

77 C4
13-2
2500X
OD
CGA

78 C1
2-2
11X
45° TO
FRACTURE
SURFACE
AIR

78 C2
2-2
110X
FRACTURE
SURFACE
AIR

78 C3
2-2
550X
ID
AIR

78 C4
2-2
2750X
ID
AIR

79 C1
2-2
100X
ID
AIR

79 C2
2-2
500X
ID
AIR

79 C3
2-2
2500X
ID
AIR

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