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QUARTERLY REPORT—JULY—SEPTEMBER 1976

GEOHERMAL MATERIALS STUDIES

Alfred Goldberg

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GEOTHERMAL MATERIALS STUDIES

by Alfred Goldberg

with contributions from

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1. INTRODUCTION

Since its inception in 1974, the LLL Geothermal Program has evolved into two basic activities:

- Providing advanced technologies for hydrothermal systems with emphasis on the total flow process.
- Providing technical assistance to joint ERDA/industry projects through the Industrial Support Program.

The Metallurgy Division is carrying out a materials effort in support of both activities.

In the total flow process, the enthalpy contained in the hot fluid at the wellhead is converted into kinetic energy by expanding the total flow from the wellhead through converging/diverging nozzles to produce low-temperature, high-velocity streams of two-phase fluid (liquid plus gas) to drive impulse turbines. This process is expected to be as much as 60% more efficient than either the flashed-steam or binary-cycle process, in both of which a large fraction of the enthalpy is lost in the relatively hot brine that is rejected. The goal for the Total Flow Process Program is to solve the brine chemistry and materials problems in order to be able to test this process using the high-temperature, high-salinity brines of the Salton Sea Geothermal Field. This particular resource was chosen because it represents the greatest technical challenge and economic potential.

The short-range goal of the geothermal materials group is to select structural materials and fabrication processes for a 100-kW, brine-tolerant turbine scheduled to be field-tested by the end of FY 1977. The materials selected must be resistant to corrosion from the high-salinity acid brines and to erosion and erosion-assisted corrosion that may result from continuous impact of the high-velocity brine droplets and entrained particles. The turbine material must be capable of supporting high sustained stresses due to

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centrifugal forces without being subject to delayed failure by stress corrosion cracking or corrosion fatigue. Cost effective materials must also be selected for the inlet lines, turbine chamber, and handling and discharging of the effluent. Candidate materials for the various components will be evaluated through an integrated program of laboratory activity and field testing.

In the Industrial Support Program, we will monitor corrosion during operation of the San Diego Gas and Electric Company's (SDG&E's) Geothermal Test Loop Facility. Other candidate plant materials will also be tested for their corrosion resistance to provide a basis for substitution should present materials prove to be inadequate. In addition, selected materials will be tested to provide corrosion information for long-range alloy development of economical, corrosion-resistant geothermal structural materials. Materials failures that arise in the operation of the SDG&E Facility are being examined and evaluated.

2. TOTAL FLOW PROCESS

2.1 Erosion Studies of Nozzle and Turbine Materials

A number of metallic materials (e.g., Ti-, Ta-, Co-, Ni-, and Fe-base alloys) are being considered for nozzle and turbine wheel components. It is expected that some of these materials will show acceptable corrosion rates on exposure to the Niland brine under normal flow conditions. However, the severe abrasive environment anticipated for the turbine system, especially that due to the high-velocity nozzle exhaust fluid, could readily destroy the protective films responsible for the corrosion resistance of such materials and thus lead to high wear rates from erosion and erosion-assisted corrosion. It is unlikely that a material will be obtained that will show adequate resistance to both erosion and stress corrosion, and therefore some type of composite approach will probably have to be used. Alternatives that we are considering to provide the required combination of structural strength and resistance to erosion and erosion-assisted corrosion are surface hardening by chemical diffusion, the use of coatings, and the use of inserts both for the turbine blades and nozzles. We are interacting with a number of industrial concerns in determining what materials to use and for production of the coatings.

Currently, the chemical diffusion studies are limited to Ti-base alloys, in particular commercial-purity Ti and Ti-6Al-4V. Coatings are being applied by several techniques, primarily on Ti-, Fe-, Ni-, Co-, and Mo-base alloys. Ceramic materials are also being considered for inserts in nozzles and, possibly, in blades. We are also examining some of the noble metals and their alloys (Au, Pt, Rh) as either substrates or coatings. Because of the unusually wide range of passivity of its oxide (Pourbaix diagram), Ta and some of its alloys are also being considered. All these materials, in the form of wedge-shaped wear plates and/or nozzle inserts, will be exposed to high velocity fluids.

The wear plates have overall dimensions of 12.70 by 44.45 by 66.68 mm (0.5 by 1.75 by 2.63 in.). The wedge edge is tapered to an included angle of 0.34 rad (20°) and a tip radius of 250 μ m (0.010 in.). The nozzle inserts are 22.2 mm in o.d. and 43.6 mm long. The erosion test nozzle and wear plate assembly is described in detail in a previous quarterly (UCID-17261-76-1). A large number of samples have been prepared, or are in the process of being prepared, for exposure. The wedge-shaped wear plate was designed to

simulate conditions expected for the leading edge of the turbine blades. It is well known that the extent of erosion varies with the angle of impact and that the weight-loss-versus-angle relationship is different for different classes of materials (e.g., ductile as compared to brittle). To examine such relationships, a variable-angle wear plate fixture was built. This fixture will support specimens measuring 51 by 51 mm and up to 3.2 mm thick (see UCID-17261-76-2). This wear-plate fixture will also provide a means to test sheet and thin plate materials that are already on hand or readily available. To date, tests have been limited to the wedge-shaped wear plates.

Chemical Diffusion Coatings

In previous quarterlies (UCID-17261-76-1 and -2) details were presented on case hardening of Ti-6Al-4V wear plates by carbonitriding and on the exposure of one of these plates for a total of 10 hours to the LLL fresh-water, high-velocity facility. Since no wear (weight loss) was detected as a result of such exposure, it appears worthwhile to further pursue the possibility of case hardening as a means to avoid erosion. Toward this end, some 80 additional wear plates and 30 additional nozzle inserts of Ti-6Al-4V were fabricated in order to develop the optimum case-hardening treatment for erosion resistance. Some of these plates have been carbonitrided. However, other priorities related to turbine design studies have prevented the evaluation of these plates at the LLL fresh-water facility this past quarter.

Hard Coatings

In previous quarterlies (UCID-17261-76-1 and -2) details were presented on a number of processes that are being used to apply hard coatings on our wear plates and nozzle inserts. These included three types of spray techniques, chemical vapor deposition, electroplating, and anodizing. Inserts and wear plates are also being made of ceramics and 3D carbon/carbon. Both coated and uncoated materials will be exposed in the field for erosion and corrosion studies in modified and expanded brines. Our current inventory of wear plates and nozzle inserts that are ready for testing is listed in Tables 2.1, 2.2, and 2.3. Wear plates and inserts that are scheduled for early delivery are listed in Tables 2.4 and 2.5.

Chemically Inert Hard Surface Films

An alternative approach to the more conventional single-layer coatings is the application of multiple layers of chemically inert, extremely hard thin films. A detailed discussion of this approach was presented in the

Table 2.1 Coated or surface-modified wear plates ready for testing. Number of samples of each type is shown in parentheses.

PLASMA SPRAY

<u>Substrate</u>	<u>Coating</u>	<u>Substrate</u>	<u>Coating</u>
Ti-6Al-4V (4)	WC/Co matrix	200 series SS ^a (1)	Al ₂ O ₃
Ti-6Al-4V (2)	WC/Co-Cd-C matrix	200 series SS (1)	Ta ₂ O ₃
Ti-6Al-4V (4)	TaC	200 series SS (1)	TaC
Ti-6Al-4V (2)	Ta-10W	200 series SS (1)	WC/Co matrix
Ti-6Al-4V (2)	Ta	200 series SS (1)	Cr
Ti-6Al-4V (2)	Cr		
Ti-6Al-4V (2)	Al ₂ O ₃		

DETONATION-GUN SPRAY

<u>Substrate</u>	<u>Coating</u>	<u>Substrate</u>	<u>Coating</u>
304 SS (3)	60Al ₂ O ₃ -40TiO ₂	Ti-6Al-4V (1)	60Al ₂ O ₃ -40TiO ₂
304 SS (1)	91WC-9Co	Ti-6Al-4V (3)	91WC-9Co
304 SS (2)	95(W+Cr)carbides-5Ni		

CHEMICAL VAPOR DEPOSITION (CVD)

<u>Substrate</u>	<u>Coating</u>	<u>Substrate</u>	<u>Coating</u>
Mo (2)	TiC	Mo (2)	WC
Mo (2)	TiC	Mo (2)	Re

ELECTROPLATING

<u>Substrate</u>	<u>Coating</u>	<u>Substrate</u>	<u>Coating</u>
Fe-26Cr-1Mo (2)	Ni	304 SS (6)	Ta
Fe-26Cr-1Mo (2)	Cr		

CHEMICAL-DIFFUSION SURFACE HARDENING

<u>Substrate</u>	<u>Coating</u>	<u>Substrate</u>	<u>Coating</u>
TZM (2)	Carburized	Ti-6Al-4V (11)	Carbonitrided
50Mo-50Re (2)	Carburized	Ti-CP40 (6)	Carbonitrided

ANODIZING^b

Illium PD (2)	CVD Rh on Mo substrate (2)
TZM (2)	CVD TiC on Mo substrate (2)
Fe-26Cr-1Mo (2)	CVD WC on Mo substrate (2)
Fe-21Cr-6Ni-9Mn (2)	CVD TaC on Mo substrate (2)
A-286 (2)	Ti-6Al-4V (2)

^a SS = stainless steel.

^b Anodizing method using proprietary additives; the process is referred to by the manufacturer as "Timadizing."

Table 2.2 Uncoated wear plates ready for testing. The metallic plates are in the as-received microstructural condition; there are two plates of each material, except some 40 plates of Ti-6Al-4V.

<u>Ti-base</u>	<u>Ni-base</u>	<u>Fe-base</u>	
Ti-CP70	Illium PD	Incoloy 833	316L SS
Ti-8Al-1Mo-1V	Illium B	Incoloy 862	18Cr-2Mo
Ti-61Al-6V-2Sn	Illium 98	Incoloy 800	21Cr-6Ni-9Mn
Ti-6Al-2Nb-1Ta-1Mo	Incoloy 825	A-286	
Ti-6Al-4V	Inconel 600	2-1/4Cr-1Mo	
Ti-1.5Ni	Hastelloy G	Fe-26Cr-1Mo	
Ti-38 (Code 12)	Hastelloy C-276	Carpenter 20CB-3	
	Nickel	200 series SS	
<u>Co-base</u>	<u>Mo-base</u>	<u>Others</u>	
Haynes 25	Molybdenum	Berylco 25	
<u>Nonmetallic</u>			
TiB ₂	AlN		
Si ₃ N ₄	3D carbon/carbon		

Table 2.3 Nozzle inserts ready for testing. Number of each type is shown in parentheses.

<u>Uncoated</u>	<u>Coated</u>
Ti-6Al-4V (8) ^a	Pt electroplated on Ni (2)
Nickel (2)	Re electroplated on Ni (2)
Gold (4)	Ta electroplated on 1018 steel (6)
Haynes 6B (2)	WC CVD on R.G. graphite (2)
Haynes 25 (2)	TiC CVD on R.G. graphite (2)
R.G. graphite (12)	SiC CVD on R.G. graphite (2)
3D carbon/carbon	TaC CVD on R.G. graphite (2)
	Ti-6Al-4V anodized (Timadized) (2)
	Haynes 25 anodized (Timadized) (2)
	Haynes 6B anodized (Timadized) (2)

^a Four of these are complete nozzles.

Table 2.4. Wear plates in fabrication. Number of each type is shown in parentheses.

Solid Metal

Inconel 625	(16)	Fe-26Cr-1Mo	(16)
18Cr-2Mo steel	(6)	PH 13-8Mo	(16)
316L SS	(16)	PH 15-7Mo	(16)
410 SS	(16)	5Cr-1/2Mo steel	(16)
430 SS	(16)	9Cr-1Mo steel	(16)
317L SS	(16)	Ti-CP30	(16)

Ceramics

TiB ₂	(2)	B ₄ C	(3)
Si ₃ N ₄	(2)	AlN	(2)
		Al ₂ O ₃ + TiC	(3)

Electroplated Coatings on Nickel Substrate

(a) Coating of 5-μm diamond particles in Ni-alloy matrix	(2)
(b) Same as (a) but heat-treated	(2)
(c) Coating of SiC particles in Ni-alloy matrix (NYE-CARB)	(2)
(d) Same as (c) but heat-treated	(2)

Table 2.5 Nozzle inserts in fabrication.

1008, 1055, and 8645 AISI steels anodized (Timadized), two of each
Ta and Zr, six of each

previous quarterly (UCID-17261-76-2). Preliminary studies of the layered film deposits will be made using flat plates of Ti-6Al-4V as the substrate. Some 60 plate samples are presently being coated for evaluation. The films will be evaluated for adherence and hardness; selected samples will be tested for erosion resistance using the variable-angle wear plate fixture.

Samples from Scaling Studies

A series of Ti-6Al-4V nozzles and wear plates (17 of each) was exposed at the LLL geothermal field unit to chemically modified brine from Magmamax Well No. 1. Exposure time was 20 hours for each run, in which modified brine expanded in the nozzle and impinged on the leading (wedge) edge of the wear plates (described in quarterly UCID-17261-76-1). The primary purpose of these runs was to examine the effect of pH (HCl additions) on scaling. The pH ranged from 1.5 to 5.7 (unmodified pH). Acidification resulted in virtually eliminating the scaling. The scaling results will be described in a separate report by L. B. Owen.

A cursory visual examination of all exposed nozzles and wear plates did not reveal any indication of erosion or corrosion. Three nozzles (exposed to pH 2.4, 3.9, and 5.6) were examined in more detail. All visible scale was removed from each nozzle throat with a hard plastic scraper. The original throats had been machined to a diameter of 6.375 ± 0.025 mm, and all three still measured within this tolerance. The leading edges of six of the wear plates (exposed to pH of 1.5 to 5.6) are in the process of being examined for erosion using a comparator technique. A number of nozzle and wear-plate sections are presently being prepared for detailed metallographic examination.

2.2 Stress Corrosion Cracking (SCC)

High static stresses will be developed in the rotating turbine wheel due to the centrifugal forces, which are proportional to the material density. On the basis of a comparison of the strength-to-weight ratios of various materials and their behavior in chloride-containing environments, Ti-base alloys appear to be the favored materials for turbine applications in the geothermal field environment. Static stresses of the order of 350 MPa (51,000 psi) are estimated for a Ti-alloy turbine wheel. Superimposed will be cyclic stresses arising, in part, from the cyclic impact of the exhaust from the nozzles. Therefore, the effect of the environment in limiting the material to withstand either static or cyclic loading must be considered.

In cyclic loading, the effect of a corrosive environment is to cause a continuous drop in fatigue strength — relative to that obtained in an inert environment — without the appearance of a well-defined fatigue limit. Under a static load, failure in a corrosive environment may occur at a stress level considerably below the yield strength of the material in an inert environment. Both of these phenomena should be similarly affected by similar environments; but since studies of static (noncyclic) loading are more widely documented, we will address the question of static loading as an illustration of the problem. Needless to say, in the final choice of materials, both static and cyclic testing must be done; however, for the initial screening process of many materials we have elected to use the much simpler static loading techniques.

The failure which occurs under the combined action of a static stress and corrosion is referred to as stress corrosion cracking (SCC). It does not take place if either the stress or the corrosion act independently. It occurs predominantly in alloys and seldom in pure metals. Stresses may be due to applied loads or to the wedging action of corrosive products, or they may be residual stresses. One of the more insidious characteristics of SCC is that frequently the alloy is virtually, or almost, inert to the environment in which SCC occurred. For example, trace quantities of chloride in drinking water can cause failure, within several hours, of most stainless steels loaded to somewhat below their yield strength. In general, the susceptibility of a given material to SCC refers to a specific environment. Frequently, it is the presence of only trace amounts of some constituent in that environment that causes the damage, i.e., different alloys or groups of alloys are susceptible to different environments, and furthermore this susceptibility will vary with even minor changes of a given environment. The pH, temperature, and oxidizing potential are also important parameters. Thus, although it may be advisable for economic or logistic reasons that initial screening tests on various candidate materials be performed in a simulated environment, it is extremely important that the final selection of materials be based on their behavior under exposure to the actual service environment.

The literature abounds with examples of industrial failures, laboratory test results, and mechanistic interpretations related to SCC. Such information may have to be used as a first-round guide in the selection of candidate materials for service in what may appear to be a similar environment. One

cannot state a priori that the final choice will be among these materials, for final testing must be performed in the actual environment. This is especially true for geothermal brines, for the corrosiveness of different brines can be expected to vary considerably. Information available on chlorides is related largely to seawater compositions, boiling $MgCl_2$ solution, and to a lesser extent various other uncontaminated chlorides; neither the pH nor the oxidizing potential is usually controlled or specified. Other constituents usually present in geothermal brines, such as H_2S and CO_2 , are most likely absent in the chloride solutions for which SCC data is available.

Very little information is available on SCC in geothermal brines. Since such chloride-containing solutions are among the most aggressive environments causing SCC, the need for extensive preliminary screening of materials is obvious. Using the information available in the literature and consulting with experts in industry and other laboratories, we have selected a number of Ti-, Fe-, Ni-, and Co-base alloys as possible candidate materials for SCC experiments in our laboratory and in the field.

Laboratory SCC Experiments on Ti-Base Alloys

In the previous quarterly (UCID-17261-76-2) we reported that no evidence for SCC was seen in any of the Ti-base alloys that were exposed to four-point, bent-beam loading in simulated Niland brine at $105^\circ C$. Two Ti-6Al-4V samples were exposed up to 2275 hours, and five other alloys were exposed up to 1363 hours. The samples had been pre-notched and loaded. Following immersion of the loaded samples, they were scribed along the notch to expose fresh surface. In addition, during the final 900 hours, samples periodically were plastically deformed in solution to a total outer-fiber plastic strain of about 10 to 15%.

Because of the apparent failure to detect any susceptibility to SCC with bent-beam samples, we discontinued this technique. In its place we have adopted a method involving constant-strain-rate tensile deformation to failure, whereby fresh metal surface is continuously being exposed at slip offsets forming at the surface. Samples are deformed at a series of strain rates. At high strain rates, there is insufficient time for corrosion to take place. At very low strain rates, the rate of surface healing (oxidizing) should be greater than the rate of surface disruptions caused by the slip offsets. It may be expected that, at some intermediate strain rate, initiation

of SCC at the slip offsets would occur, resulting in a reduction of mechanical products in the brine relative to those obtained in a noncorroding medium. For Ti-8Al-1Mo-1V, a drop of approximately 50% in elongation and tensile strength is obtained in 3% neutral NaCl solution at room temperature at a strain rate of about 10^{-5} s^{-1} (private communication with Neville Pugh, University of Illinois).

Tensile sheet samples were machined from some of the remaining unused bent-beam sheet samples of Ti-6Al-4V, Ti-6Al-4V ELI, Ti-3Al-8V-6Cr-4Mo-4Zr, and the control alloy Ti-8Al-1Mo-1V. These were deformed in the as-received conditions in a simulated brine solution, using an Instron machine.* The experimental setup is shown in Fig. 2.1. To maintain relatively reducing conditions, the brine solution was continuously purged with argon at a rate of about $0.15 \text{ m}^3 \cdot \text{h}^{-1}$. The solution was maintained at a pH of 3 by HCl additions and held at 100°C .

The brine solution was based on a Sinclair No. 4 Well analysis. The following was added to a basic brine solution of 2.8M NaCl-0.41M KCl-0.73M CaCl_2 : 32 ppm S^{2-} (as $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$), 1100 ppm Mn^{2+} (as $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$), 1680 ppm Fe^{2+} (as $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), and 80 ppm Mg^{2+} (as $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$). The S^{2-} content would continually decrease by the escape of H_2S .

Samples were deformed in the brine at tensile strain rates of 2.50×10^{-4} , 10.3×10^{-5} , 1.77×10^{-6} , and $4.12 \times 10^{-7} \text{ s}^{-1}$. In addition, three of the alloys were deformed at the strain rate of $1.03 \times 10^{-5} \text{ s}^{-1}$ at 100°C in air. The ultimate tensile strength (UTS) and % elongation for the four alloys are shown plotted as a function of strain rate in Figs. 2.2-2.5. No definitive evidence for SCC is indicated from the results. Some indication for SCC is perhaps suggested for the Ti-3Al-8V-6Cr-4Mo-4Zr and Ti-6Al-4V ELI alloys. The Ti-8Al-1Mo-1V alloy which should show some susceptibility to SCC does not show any indication of it at all. Of greater significance, however, are the surprising results that both the elongations and tensile strengths obtained for the air samples are lower than those observed for the brine solution samples. We wish to reserve further comments on these anomalous results pending examination of the fractures.

Fig. 2.1 Experimental setup for screening materials for SCC by the use of constant-strain-rate testing.

Fig. 2.2 Effect of strain rate on ultimate tensile strength (UTS) and elongation of Ti-8Al-1Mo-1V alloy samples in simulated Niland brine (pH = 3) at 100°C and continuously purged in argon, and in air at 100°C. Note lower values in air.

Fig. 2.3 Effect of strain rate on strength and elongation of Ti-6Al-4V alloy samples in simulated Niland brine (pH = 3) at 100°C and continuously purged with argon, and in air at 100°C. Note lower values in air.

Fig. 2.4 Effect of strain rate on strength and elongation of Ti-3Al-8V-6Cr-4Mo-4Zr alloy samples in simulated Niland brine (pH = 3) at 100°C and continuously purged with argon.

Fig. 2.5 Effect of strain rate on strength and elongation of Ti-6Al-4V ELI samples in simulated Niland brine (pH = 3) at 100°C and continuously purged with argon, and in air at 100°C. Note lower values in air.

In addition to the above samples tested, we have more recently obtained some 20 tensile samples of each of the following alloys: Ti-8Al-1Mo-1V, Ti-6Al-4V, Ti-6Al-4V ELI, Ti-6Al-4Zr-6Mo, Ti-3Al-8V-6Cr-4Mo-4Zr, Ti-15V-3Cr-3Al-3Sn, and Ti-4Al-3Mo-1V. It was our intent to screen these alloys in several heat-treated conditions using the above constant-strain-rate technique. We have delayed these additional tests until we obtain a satisfactory solution to the dilemma presented in the current results.

Laboratory SCC Experiments on Some Fe-, Ni-, and Co-Base Alloys

Nine Fe- and Co-base alloys, which are part of an extensive series of Fe-, Ni-, and Co-base alloys to be field-tested (see Field Test Section below) were tested in the form of U-bend samples in simulated, pH 3 brine at 100°C. The experimental setup is shown in Fig. 2.6. The brine was prepared as described in the previous section; however, the solution, instead of being purged with argon, was continuously aerated, since such aeration should accelerate attack of these materials. The samples measured 152 mm long by 15.9 mm wide by approximately 1.6 mm thick. They were final-polished with 320-grit emery paper. All materials were in the mill-annealed condition.

To obtain additional information on crevice corrosion in stressed samples, we bent two samples of each alloy together to a U-shape in a press over a 25.4-mm-diam mandrel and bolted them together in place. Thus, the outer sample was exposed to the brine in tension and to the crevice condition in compression, with the reverse conditions for the inner sample. Two such sets of samples were exposed, i.e., four of each alloy, one set for 500 hours, the other for 614 hours. The alloys tested are listed in Table 2.6. The relative ranking of these alloys based on a preliminary examination with the stereomicroscope is given in Table 2.7. The solution level was maintained just above the curved portion of the U-bend. Damage was primarily in the form of pitting and in the development of fissures or cracks on exposure to the brine liquid (stressed portion of sample) and pitting on exposure to the brine vapor (relatively unstressed portion). Relatively thin, adherent oxide films had formed on all external surfaces to various degrees. However, the two inner surfaces in contact with each other in each case remained mostly clean and bright except along the edges and where pits had formed. The pits on these inner surfaces are referred to as crevices. The alloys are ranked primarily on the basis of pitting in the liquid phase. Haynes 25 showed the best resistance to attack and was the only alloy which did not show any evidence of pitting,

Fig. 2.6 Experimental setup for testing U-bend samples.

Table 2.6 Composition (wt%) of alloys exposed to aerated simulated brine.

Alloy	Cr	Ni	Mo	Mn	C	Other main constituents	Crystal structure
Haynes 25 ^a	19.85	10.42	—	1.61	0.10	14.43W, 2.33Fe ^b	fcc
Alloy 6X ^a	20.30	24.30	6.54	1.69	0.02	0.05Al, 0.04Nb, 0.03N	fcc
29-4 ^a	29.40	0.11	3.95	0.02	0.005	0.6Al, 0.01N	bcc
316 SS ^c	17	13	2.5	—	—		fcc
Nitronic 33 ^a	17.83	3.34	—	13.87	0.046	0.29N	fcc
304 SS ^c	18	10	0	—	—		fcc
Alloy 439 ^c	18	—	—	—	—	Ti-stabilized	bcc
Fe-26Cr-1Mo	25.59	0.11	1.03	0.01	0.0010	0.0010N	bcc
18 Cr-2Mo ^c	18	—	2	—	—	Ti-stabilized	bcc

^a Actual.

^b Co-base; all others Fe-base.

^c Nominal.

Table 2.7 Relative ranking of the corrosion-resistant alloys of Table 2.6 as determined by the exposure tests. Based on visual and stereomicroscopic (up to 32×) examination of U-bend specimens exposed in aerated simulated brine. Two series of exposures, 500 and 614 hours. A ranking of 1 means most resistant, 9 means least resistant.

Alloy	Brine liquid exposure			Brine vapor exposure	
	Pitting	Fissures	Crevice	Pitting	Crevice
Haynes 25	1	1	1	1	1
Alloy 6X	2	3	3	2	1
29-4	3	2	2	6	5
316 SS	4	6	4	3	3
Nitronic 33	5	5	5	4	4
304 SS	6	7	9	5	9
Alloy 439	7	— ^a	7	7	7
Fe-26Cr-1Mo	8	— ^a	8	9	8
18Cr-2Mo	9	4	6	8	6

^a Pitting obscured identification of fissures.

crevice corrosion, or stress corrosion cracking. The most severely attacked of these alloys was 18Cr-2Mo-Ti-stabilized. In view of the promising results reported for 26Cr-1Mo by Needham for geothermal environments, we were surprised with the poor performance shown here for this material; yet, our own experience with 26Cr-1Mo tubing in the field is consistent with our laboratory results. Examination of these specimens will be continued to identify more clearly the presence and nature of stress corrosion cracks. In many cases, the presence of cracks was obscured by excessive pitting; the term fissure as used in Table 2.7 implies cracks and/or pitlike channels. Figure 2.7 contains macroscopic views of Haynes 25 and 18Cr-2Mo-Ti-stabilized showing the two extreme examples. Apparent "defects" seen on the surface of the Haynes 25 alloy are patches of oxide films.

Field Test of a Ti-Base Alloy

Two sets of Ti-6Al-6V-2Sn alloy samples were exposed with a wear plate to one of the brine-modification scaling tests. The samples were mounted in a bent-beam four-point-loading configuration, as described in a previous quarterly (UCID-17251-76-1), i.e., two bend-constrained overlapping samples in contact with each other, one pair on each side of the wear plate. The samples were exposed for 20 hours to the modified expanded brine with a pH of 3.2. The exposed assembly including the nozzle exit, the wear plate, and one pair of samples is shown together with the wear plate attached to the fixture in Fig. 2.8. The purpose of attaching these bent-beam specimens in this test was to determine whether backscattering of the brine would interfere with the scaling experiment; it did not. Although this alloy is not being considered as one of the more likely Ti-base candidates, we nevertheless thought that examining the exposed samples would be worthwhile.

Some scale did form on the samples (although the amount of scale formed on the wear plate was negligible). This scale was removed from one pair of samples which were then examined with the stereomicroscope; no visible evidence of corrosion or cracks could be detected. The second set was mounted, polished, and etched for microscopic examination. The upper surface of the sample exposed to the direct impact of the flow contained numerous indentations up to about 10 μ m deep. By contrast, the surface in contact with the underlying sample and both surfaces of the underlying sample were relatively smooth. The indentations can be seen in Fig. 2.9. We believe that these indentations are the result of erosion.

Fig. 2.7 U-bend samples after exposure to simulated Niland brine for 614 hours. Brine was controlled to a pH of 3, aerated, and kept at 100°C. (a) Haynes 25, (b) 18Cr-2Mo-Ti-stabilized. Left-hand sample of each material is the "protected" inner sample, right-hand sample is the "exposed" outer sample.

Fig. 2.8 Wear-plate/nozzle assembly with bent-beam samples after 20-hour exposure to chemically modified Niland brine ($\text{pH} = 3.2$). Pattern on wear plate reflects flow characteristics of nozzle exhaust and deposition of fine mixed deposit of scale and salt.

Fig. 2.9 Surface appearance of Ti-6Al-4V-2Sn bent-beam samples exposed for 20 hours to chemically modified Niland brine (pH 3.2) in wear-plate/nozzle assembly of Fig. 2.8. (a) Surface in direct path of nozzle exhaust is badly eroded. (b) Surface of underlying sample is not eroded.

High-Strength Fe-, Ni-, and Co-Base Alloys

Screening studies are planned to investigate the susceptibility to SCC (as well as to other forms of corrosion attack) of several high-strength Fe-, Ni-, and Co-base alloys. The alloys chosen are MP35N, Haynes 25, Hastelloy C-276, Inconel 718, PH A286, PH 13-8Mo, and PH 15-7Mo. In order to achieve high strengths, all but the last two of these alloys must be cold-worked before aging. These alloys have been received in plate form and are being reduced to various thicknesses and cold-rolled conditions by the Bureau of Mines in Albany, Oregon, for subsequent fabrication into corrosion coupons, corrosion electrodes, and SCC sheet specimens. Final heat-treatment and preparation of specimens will be done at LLL.

2.3 General Corrosion Field Tests

In addition to the nozzles, turbine blades, and turbine wheel we will be concerned with corrosion of the turbine case, the turbine exhaust system, effluent reinjection lines, pumps, valves, piping, and well casings. We will therefore examine a number of materials that may be considered as candidates for these various components. Results to be obtained from corrosion studies for the Industrial Support Program described in a later section will provide some useful information for the turbine system. However, such results must be used with caution since the corrosion behavior in the modified brines may be quite different from the behavior indicated by results obtained in unmodified brine. We are therefore planning to examine various materials exposed to the effluent from the modified brines.

As a first series of screening tests, the 102-mm-diam, 610-mm-long discharge elbows just downstream from the three nozzle-wearplate test stations at the LLL field unit will serve as test specimens. We will examine carbon steels including typical API steels, a series of Cr-Mo steels, stainless steels, titanium of commercial purity, and some titanium alloys. Along any given cross section, the elbow will be subjected to a continuous variation in erosion/corrosion conditions which should become more damaging as one goes from the inner to the outer elbow radius. Turbulence of the flow will also vary on passing from inlet to outlet. Thus, the elbow specimens should offer an excellent means for screening materials in the chemically modified brines over a wide range of flow conditions.

Seventeen AISI 1018 steel elbows which were exposed to the brine-modification scaling experiments are being examined. The scale has been removed from five of these elbows (exposed to pH 1.5 to 5), and they are in the process of being sectioned for visual examination and measurement for evidence of erosion. A number of the remaining exposed elbows have been mounted for metallographic observations of scale deposits (by Owen). The elbow samples will subsequently be used for evaluation of corrosion and erosion.

Elbows are presently being fabricated from 4130 steel, 304 stainless steel, and Fe-26Cr-1Mo alloy. Tubes from commercial-purity Ti, Ti-6Al-4V, ingot iron, COR-TEN, 317L stainless steel, and 316 stainless steel are on hand, ready for fabrication into elbows. Plate stock of MP35N is also available for fabrication into tubes and then into elbows. In addition to testing materials in the form of elbows, we also plan to expose a wide range of materials to the modified brines in the form of corrosion coupons and corrosion electrodes. Additional reference to these two types of specimens is made in the following section describing the Industrial Support Program.

3. INDUSTRIAL SUPPORT PROGRAM

3.1 Evaluation of Corrosion and Erosion Failure

An important aspect of the Industrial Support Program is surveillance of corrosion problems. This involves the repeated inspection of plant components in service, the analysis of failures, and the recommendation for replacement parts and/or design modification. Several reports and papers have been published, and others will be published, as a result of these analyses. The discussion of potential problems related to the stainless steel claddings (see January-March Quarterly, UCID 17261-76-1) has now been published in a separate report ("Comments on the Use of 316L Stainless Cladding at the Geothermal Niland Test Facility," UCID-17113). A paper on the analysis of failures due to pitting corrosion of Sinclair Well No. 4 surface piping and a casing liner is presently being prepared for presentation at the October Electrochemical Society Meeting and for subsequent publication. (See April-June Quarterly, UCID-17261-76-2, for details of the failures.) Field failures from SDG&E, which are currently being examined, involve well-pressure sensing tubing, two elbows, a valve, and two rotating shafts. These are discussed below.

Elbow and Valve Failures at SDG&E Test Loop Facility

A 152-mm-diam (6-in.) elbow, located immediately downstream from the control valve for the first-stage flash drum, had been perforated along the outer radius as a result of approximately 150 hours of intermittent operation of the SDG&E Geothermal Test Loop Facility. The brine, flowing at about $180 \text{ Mg} \cdot \text{h}^{-1}$, was supplied from the Magmamax No. 1 well. The elbow had been badly eroded as revealed by extensive gouging. The perforation was patched by welding on to the outside surface a plate measuring 240 by 150 mm. The gouges were filled with weld metal. The elbow was made of A-106 plain carbon steel. The repaired elbow was not put in use but instead was sent to LLL, arriving on June 7. An attempt was made to analyze the elbow using radiography and metallography. However, the roughness of the interior surface of the elbow and the voids in the wall which were introduced by the filler metal made the analysis difficult, and it was discontinued. Erosion rather than corrosion was concluded to be mainly responsible for the failure. Figure 3.1 shows a view of the elbow and patch. Figure 3.2 is a view looking down into the exit end of the elbow. Much of what is seen is due to a mixture of scale and splattered filler metal. A composite of longitudinal cut sections is

Fig. 3.1 This 152-mm-diam elbow was perforated during 150 hours of intermittent operation at the SDG&E Geothermal Test Loop Facility. Note welded-on patch plates.

Fig. 3.2 View into inlet end of perforated elbow of Fig. 3.1. Patch plate is hidden from view.

shown in Fig. 3.3. The cut surfaces were ground, and they revealed that the inside surface along the outer radius of the elbow in the area under the patch plate was quite rough as compared to the surface of the rest of the elbow. Macroetching showed that this roughness was associated with the filler metal. In Fig. 3.3 one can also see erosion markings in the scale and in the metal surface where the scale had flaked off; these markings can be related to the flow direction.

On June 23, 1976, the elbow which had been used as a replacement for the above-mentioned elbow was recieved at LLL. Together with this elbow, we received a control valve which was badly eroded. The valve was dismantled and is presently being analyzed. Figure 3.4 shows a view of the valve body and a connection flange.* A composite of two longitudinally cut sections of the elbow is shown in Fig. 3.5. One may note that significant loss of metal (thinning of the wall) had occurred along a section of the inside wall at the outer elbow radius. The analysis of wear in these two parts is being continued. Again, we believe the problem is mainly due to erosion.

Shaft Failure at SDG&E Test Loop Facility

Two shafts arrived at LLL from SDG&E in early September 1976. Both had failed during operation of the SDG&E Test Loop Facility. One shaft, 35 mm in diameter, came from a vacuum pump. The fracture surface showed a smooth transverse break; no corrosion was evident. We concluded that the failure was the direct result of an overload.

The second shaft, 51 mm in diameter, came from a nine-stage reinjection pump. The surfaces of the original fracture were largely destroyed. Seizure and galling of the shaft at bearing surfaces were clearly indicated, and this suggested that failure had occurred due to torsional overload. Visual examination indicates that mutual rubbing of the fractured surfaces created significant heat to melt part of the metal. The roughness, apparently caused by such localized melting, precludes analysis of the fracture surface. If time permits, we may section the fractured end to see if any additional information can be obtained.

* The two exposed surfaces were mating surfaces when the first elbow was in service. The flange was then cut off and welded onto the exit end of the second elbow, as shown in Fig. 3.5.

Fig. 3.3 Composite of longitudinally cut sections of elbow of Fig. 3.1. Note erosion striations.

Fig. 3.4 Central valve body and connecting flange. Matching surfaces are badly eroded.

Fig. 3.5 Longitudinal sections of replacement elbow, extension pipe, and flange. Note thinning of wall at location A.

Failure of Downhole Pressure-Sensing Tubing

Pressure-sensing tubing made of ferritic stainless 26Cr-1Mo failed after being submerged for one month in the Magmamax No. 3 Well. This well was used as a reinjection well for the SDG&E geothermal test loop facility. The tubing, which was removed from the well on May 27, 1976, has a 2.44-mm o.d. and a wall thickness of 0.76 mm. It supported a downhole pressure chamber weighing 19 kg. The tubing had broken off at a depth of 396 m in the well. Sections of the failed tubing were sent to us for examination. The tubing was extensively pitted; microstructural examination indicated that the tubing was in a corrosion-sensitized condition. This could be due to improper final heat treatment or to carbon or nitrogen pickup occurring during thermomechanical processing of the tubing.

Details of a preliminary analysis of the failure were reported in a Metallurgy Division Technical Note (MD-01, June 24, 1976), in which we described the microstructure of the tubing, presented possible reasons for its failure, and suggested the use of alternative materials, namely MP35N, Hastelloy C-276, E-Brite 29-4, and Ti-Code-12, among others. We also recommended that these materials should be field-tested.

Figure 3.6 shows an enlarged view of a section adjacent to the failure at the 396-m depth in the well. The fracture resulted from a simple bending of the tubing as shown, indicating its brittleness. Numerous pits can be seen. Longitudinal and transverse polished sections are shown in Fig. 3.7. Surprisingly, pitting was primarily restricted to a circumferential region extending about 1.5 to 2.0 radians around the tube. Figure 3.8 shows etched areas of pitted regions taken near a fracture that had occurred in rewinding the tubing, at a point corresponding to a 209-m depth. The sawtooth attack shown in view (b) suggests that the corrosion potential is close to the region where stress corrosion cracking could occur. What follows is a continuation of effort since writing the MD-01 technical note.

Over 5000 m of the 26Cr-1Mo tubing had been obtained for the SDG&E operation. It was therefore important to determine what action could be taken to salvage the remaining unexposed material. We have been in contact with the company where the tubing was processed and also with representatives of the producers of the alloy. It was felt that nitrogen contamination may have resulted from the nitrogen quench used in each of the seven annealing treatments during reduction of the tubing to its final size. Carbon infusion

Fig. 3.6 Numerous deep corrosion pits are evident along this brittle portion of 26-Cr-1Mo tubing that was close to the point where failure occurred at a depth of 396 m in the well. This sample cracked on initial bending.

Fig. 3.7 (a) Longitudinal and (b) transverse polished sections of the tubing shown in Fig. 3.6. Note in (b) that pits are present only on a portion of the circumference (1.5 to 2.0 radians).

Fig. 3.8 Longitudinal section of 26Cr-1Mo tubing exposed at the 209-m level in the well, corresponding to the portion of tubing that failed during rewinding after removal from the well. (Electrolytic etch in 40% HNO_3 -60% H_2O .)

from the drawing lubricants was a second possibility. If the material was not contaminated but had simply suffered from a poor thermal history, then it should be salvageable by proper heat treatment.

Lengths of both corroded and unexposed samples of the 26Cr-1Mo tubing were sent to the alloy producer for examination. However, shortly thereafter, we were notified that a decision had to be made on downhole use of some 300 m of the tubing which was then sent to us. Following consultation with the alloy producer, we carefully cleaned the tubing in MEK and sent it to Electrofusion Corporation for heat treatment. This consisted of vacuum-annealing the coiled tubing for 30 min at 871°C and then rapidly heating it to 982°C, keeping it at this temperature for 6 minutes, and then argon-quenching it. The temperature dropped to 570°C in 15 min and to 276°C in an additional 25 min. The tubing had been wound on a spool which had been made from perforated stainless steel sheet in order to maximize circulation of the gas during cooling. In late August, we were notified that this annealed tubing had failed after two weeks in service. We recommended that, pending further investigation of this failure, an attempt be made to obtain MP35N, Hastelloy C-276, or Ti-Code-12 tubing.

A piece of the original unexposed 26Cr-1Mo tubing which we had sent to the alloy producer was heat-treated by them according to their recommendations given to us. They evaluated the original and heat-treated conditions; the results are listed in Table 3.1. According to ASTM A268 the specifications for carbon and nitrogen are 100 and 150 ppm maximum, respectively; oxygen is not given. The alloy producer's representatives claim that no detrimental effects should be obtained below 200 ppm N. They list a typical 26Cr-1Mo alloy with 10 ppm C and 100 ppm N. We had Metallurgical Labs Inc. analyze two pieces that had been removed from the 300-m length of tubing before and after annealing. The values reported are 20 ppm C and 140 ppm N prior to annealing and 30 ppm C and 140 ppm N after annealing. Although the carbon contents are on the high side, both the carbon and nitrogen contents are well within the allowable limits. The material is indicated as being properly heat-treated if it shows a Streicher corrosion rate test value of less than 0.002 ipm (51 μ m per month). The sample that was heat-treated by the alloy producer, as shown in Table 3.1, did pass this test. The question now raised by us is whether there was an important difference in cooling rates between the alloy producer's and the Electrofusion heat treatments. We are presently looking into this

Table 3.1 Results reported by the alloy producer on original (unexposed) samples of SDG&E's Fe-26Cr-1Mo alloy pressure-sensing tubing, before and after application of the heat treatment recommended by the alloy producer.

	Before heat treatment	After heat treatment
Streicher corrosion rate test (ipm)	0.00557	0.00139
Weld hardness, R_B	99.6	93.4
Base metal hardness, R_B	98.9	85.4
Tensile strength (psi)	94,000	62,000
0.2% offset yield strength (psi)	75,700	47,000
Elongation (%)	2.5	<22.5
Carbon content (ppm)	6.0	—
Oxygen content (ppm)	202	—
Nitrogen content (ppm)	131	—

NOTE: The ingot analysis is given as C < 10 ppm, O = 58 ppm, N = 115 ppm. Gibson Tube Company sample made from the same heat gives C = 7 ppm, O = 79 ppm, N = 118 ppm.

question. We have asked SDG&E to send us sections of the newly failed 300 m of tubing.

3.2 Corrosion Testing

It was expected that in the surveillance of materials problems during the operation of the SDG&E Geothermal Test Loop Facility, especially during its early stages of operation, failures of some plant components would take place, and this has happened. It is also likely that unacceptable corrosion rates of other more lasting components will become evident. Some of the problems may be overcome by design changes; in other cases, materials with superior corrosion and or erosion resistance will be required. To assure that proper substitute materials are specified, information must be made available on the relative corrosion resistance of materials in these high-salinity brines. Initially, preliminary information should at least be obtained on readily available commercial materials for rapid replacement of failures. But of more lasting importance, we must also address the choice of plant materials from an economical, long-range point of view, and toward this end a more extensive range of materials will have to be tested. In particular, tests should be performed which would provide information for evaluating the effects of such alloying elements as Cr, Mo, and Ni, individually and synergistically, in low- and medium-alloy steels and in corrosion-resistant materials. The program we have initiated addressed both of these tasks - short-range screening tests for replacement materials, and long-range alloy development studies.

Present Status of Test Program

For low-cost replacement materials, we have fabricated a number of corrosion spools out of 0.27-m-diam pipe of different API steels for insertion in one of the supply lines to the SDG&E Geothermal Loop Facility. Details are given in the previous Quarterly (UCID-17261-76-2). Arrangements for incorporating these corrosion spools in the line are still pending.

We are planning to expose a large number of alloys as corrosion coupons in a bypass loop located between Magmamax No. 1 Well and the LLL Geothermal Field Test Unit. The loop consists of three pipes in parallel, 2.6 m long by 76 mm in diameter. Racks for suspending the corrosion coupons in the loop have been fabricated. Each rack can hold 112 flat specimens 25 mm by 51 mm by approximately 3 mm thick. The alloys being used for corrosion coupons will also serve for corrosion rate measurements and possibly polarization studies

in the field. The corrosion electrodes measure approximately 44 mm long by 6.4 mm in diameter. Details of the electrochemical corrosion tests are given in the previous Quarterly (UCID-17261-76-2). Finally, some of the higher strength alloys will be used for SCC tests; these specimens measure approximately 1.6 mm thick.

The various series of alloys to be tested in the field are listed in the previous Quarterly (UCID-17261-76-2). These alloys are presently being reduced by rolling to appropriate thicknesses for specimen fabrication. Since we plan on correlating the results obtained from the different types of tests, it is important that the different series of samples of a given alloy be taken from the same stock and subjected to the same final thermomechanical treatment. Most of these alloys have been reduced to the required thicknesses and are ready for machining into specimens.

Electrochemical Measurements

On August 11, 1976, between 1100 and 1415 hours, two redox potentials of cooled depressurized brine from the Magmamax No. 1 wellhead were taken. The brine was continuously flowed through a cooling coil into a beaker containing a Pt indicator electrode and a Ag-AgCl reference electrode. The two E_{NHE} values obtained for the two flowing solutions are +0.15 and +0.20 V. Readings changed less than 0.5 mV per min. The pH was 5.4. One would expect a more reducing brine than is indicated by these values, which are, in fact, near those observed for simulated brine at pH 5 in equilibrium with metallic Fe. No H_2S odor was evident on the day of these measurements; the absence of H_2S is consistent with the relatively high redox readings.

The two brine samples were aerated, and after 1-1/2 hours both reached a steady value of $E_{\text{NHE}} = +0.28$ V. In the past, values as high as +0.40 V have been obtained for aerated brine from Magmamax NO. 1 Well. We are somewhat concerned with the day-to-day or even hour-to-hour variations in redox potentials. This will especially be a factor in comparing results based on electrochemical measurements, such as instantaneous corrosion rate measurements or short-run potentiostatic measurements. We are therefore planning to make provisions to monitor the redox potential during extended periods of testing.

4. ELECTROLESS NICKEL PLATING OF A TURBINE WHEEL

A large turbine wheel (1.067 m in diameter) was fabricated from 7075-T6 aluminum for the evaluation of turbine performance at the LLL pressurized-fresh-water facility. Preliminary exposure tests with wear plates of this alloy showed that the turbine blades would undergo excessive wear unless their surfaces were protected by some form of coating. Of the various coatings that were considered, electroless nickel plating appeared the most promising.

Evaluation of electroless nickel-plating methods and testing of plated wear plates were undertaken. A satisfactory process and product were developed. Test wear plates, plated with nickel-1 wt% copper to a thickness of 0.075 mm, withstood the impact of water droplets at velocities of $475 \text{ m}\cdot\text{s}^{-1}$ for 3 hours. The hardness varied from 525 to 545 DPH (100-g load). Subsequently, the turbine wheel has been successfully plated and has withstood testing. Details of this work are presented in UCID-17253 by H. J. Wiesner et al.

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