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Stress Corrosion Studies in Solvent Refined Coal Liquefaction Pilot Plants

V. B. Baylor
J. R. Keiser
M. D. Allen
E. J. Lawrence

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STRESS CORROSION STUDIES IN SOLVENT REFINED COAL
LIQUEFACTION PILOT PLANTS

V. B. Baylor, J. R. Keiser, M. D. Allen,
and E. J. Lawrence

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STRESS CORROSION STUDIES IN SOLVENT REFINED COAL
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V. B. Baylor, J. R. Keiser, M. D. Allen,
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ABSTRACT

Coal liquefaction plants with 6000 ton/d capacity are currently being planned by DOE as a step toward commercial production of synthetic fossil fuels. These plants will demonstrate the large-scale viability of the Solvent Refined Coal (SRC) process, which has been used since 1974 in two operating pilot plants: a 50-ton/d unit at Fort Lewis, Washington, and a 6-ton/d plant in Wilsonville, Alabama. Experience in these plants has shown that austenitic stainless steels are susceptible to stress corrosion cracking associated with residual stresses from cold working or welding. The corrodents responsible for the cracking have not yet been positively identified but are suspected to include polythionic acids and chlorides.

To screen candidate materials of construction for resistance to stress corrosion cracking, racks of stressed U-bend specimens in welded and as-wrought conditions have been exposed at the Wilsonville and Fort Lewis SRC pilot plants. These studies have identified alloys that are suitable for critical plant applications.

INTRODUCTION

Background and Process Description

Clean-burning fuels can be produced by dissolution of coal by slurrying pulverized coal with process-derived solvent and then hydrogenating under high pressure and temperature. The coal structure is hydrocracked, resulting in a replacement of sulfur and oxygen with hydrogen to yield coal-derived liquids, acid gases, and undissolved solid particles. Various separations downstream remove the acid gases, particulate matter,

and recycle gases and liquids, leaving a low-sulfur, virtually ash-free product. The four major pilot plants using direct coal liquefaction processes are summarized in Table 1.

The Solvent Refined Coal (SRC) process has been demonstrated by two operating pilot plants: a 50-ton/d unit in Fort Lewis, Washington, and a 6-ton/d plant in Wilsonville, Alabama. There are two SRC modes, SRC-I and SRC-II (Fig. 1). In the SRC-II mode more of the dissolved coal is converted to distillate liquids and gases in the reaction area due to the increased amount of hydrocracking produced by an increased effective residence time and reaction rate.¹ The solids remaining in the process stream are part of the vacuum distillation residue which is fed to a gasifier for hydrogen production. Thus the filtration step is eliminated in SRC-II, and the main product is a liquid as opposed to the solid boiler fuel produced by SRC-I.

The Wilsonville Plant (SRC-I) was designed and constructed by Catalytic, Inc., which has operated it since 1974 under the management of Southern Company Services, Inc. The project is jointly sponsored by the Department of Energy (DOE) and the Electric Power Research Institute (EPRI). The Fort Lewis Plant, which can be operated in either the SRC-I or SRC-II mode, is being operated by the Pittsburg and Midway Coal Mining Company (P&M), a subsidiary of Gulf Oil Corporation. Sponsored by DOE, the plant was designed by P&M and built by Rust Engineering in 1974. Further details on the operating history and process conditions of the SRC pilot plants are available.^{2,3}

Two other major coal liquefaction processes, H-Coal [developed by Hydrocarbon Research, Inc. (HRI), and Ashland Synthetic Fuels, Inc.] and Exxon Donor Solvent (EDS) (developed by Exxon Research and Engineering Company) have been demonstrated successfully in bench-scale and process development units. Larger scale pilot plants have been built for both processes; however, they have just begun operation. The processes differ from the SRC process primarily in the reaction mechanism. The EDS process uses a prehydrogenated solvent to slurry the coal in addition to the hydrogen introduced before reaction. The H-Coal process uses an ebullated bed reactor with a chromium-molybdenum catalyst to increase hydrogenation of the coal. These two processes yield a higher hydrogen-to-carbon ratio than that derived from SRC, giving a more refined liquid product.

Table 1. Coal Liquefaction Pilot Plants

Process	Plant ^a	Classification	Reactor Temperature ^b		Hydrogen Pressure Range		Products
			(°C)	(°F)	(MPa)	(psi)	
SRC-I	Wilsonville, Ala. Catalytic, Inc. 6 tons/d	Solvent refining	440	825	11.7-13.8	1700-2000	Solid SRC
SRC-II	Fort Lewis, Wash. P&M Coal Mining Co. 50 tons/d	Solvent refining	455	850	10.3-13.1	1500-1900	Fuel oil Light distillate LPG ^c Pipeline gas
H-Coal	Catlettsburg, Ky. Ashland Synthetic Fuels, Inc. 600 tons/d	Catalytic hydro- genation	455	850	15.5-18.6	2250-2700	Crude oil Fuel oil Gas
EDS	Baytown, Tex. Exxon Synthetics 250 tons/d	Donor solvent	455	850	10.3-13.8	1500-2000	Gasoline blend stock (after hydro- treating) Heavy fuel oil Distillate fuel oil

^aNameplate capacity is given; 1 ton = 907 kg.

^bNominal values, actual temperatures may vary to as low as 410°C.

^cLiquefied petroleum gas.

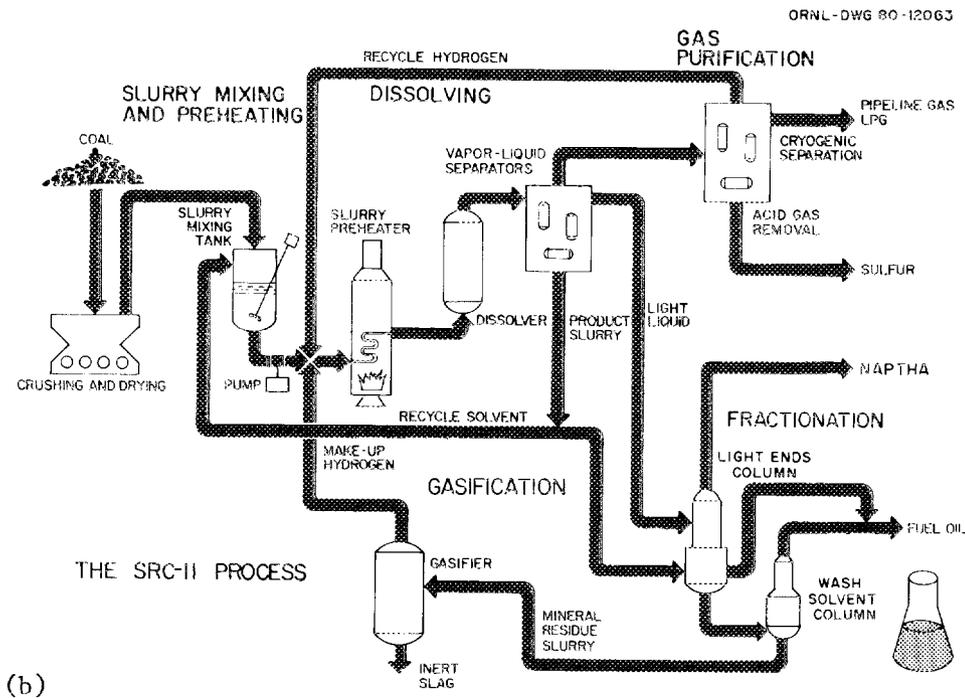
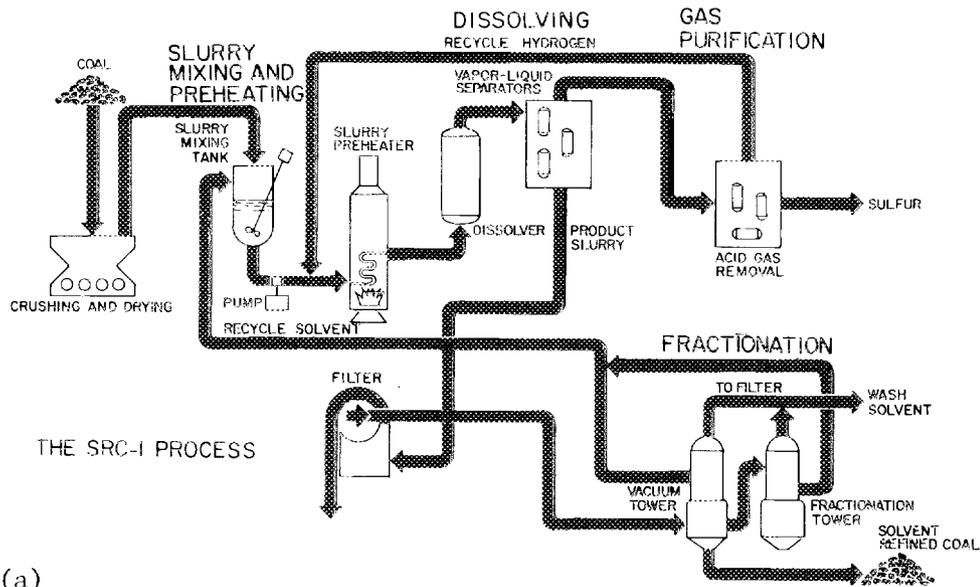


Fig. 1. Schematic Flow Diagrams of the Solvent Refined Coal (SRC) Processes. The significant differences between (a) SRC-I and (b) SRC-II are the filtration step and the melting point of the product. These differences are due to the increased hydrocracking possible in SRC-II, producing a more refined liquid product.

Although the coal liquefaction processes differ in the hydrocracking mechanism and the resultant end product, many critical regions and process streams are common among them. These are the coal preparation and slurry mixing, preheater, vapor-liquid separation, sour gas treatment, solids removal, and fractionation. Furthermore, even though the reaction mechanisms vary, there are many similarities in the reaction area (except perhaps for H-Coal), especially in terms of temperature and pressure of operation and chemical composition of vapor and liquid phases. Thus, although this report describes materials experience and testing in the SRC pilot plants, it is expected that the results will be applicable not only to the SRC demonstration plants but also to other coal liquefaction process plants.

Materials Experience

Commercialization of coal liquefaction processes depends on the reliable operation of much larger scale plants ($>20,000$ tons/d) than currently exist. Thus, good materials performance is vital to commercializing production of synthetic fuels from coal. It is not clear that the materials experience in pilot plants will have any relation to that observed in larger scale plants, but certainly problems have been encountered which appear to be more related to characteristics of the process streams rather than to design. One such problem is the stress corrosion cracking of austenitic steels which has been observed in the reaction and vapor-liquid separation areas in the SRC pilot plants.

Stress corrosion cracking failures are due to the combined action of a corrosive agent and a mechanical stress. In coal liquefaction systems, the corrosive species are most likely to be chlorides, organic acids, and, especially, polythionic and other sulfur-containing acids. Polythionic acids, $H_2S_xO_6$ ($x = 3-5$), are formed by exposure of metal sulfide scales to moist air during shutdowns. Stresses can result from applied loads but can also be residual, as produced by welding or mechanical deformation during metal processing and assembly. Cracking may be intergranular or transgranular; the former is often caused by polythionic acid, while the latter is often attributed to chlorides.

Stress corrosion cracking has been observed at both the Wilsonville and Fort Lewis SRC pilot plants. Nondestructive examination of the Wilsonville dissolver was performed in July 1977, using dye-penetrant and metallographic replication techniques. The dissolver vessel is cast type 310 stainless steel (CK 40) to which a type 347 stainless steel flange is welded. The flange cover plate (or dissolver head) is also type 347 stainless steel. Two type 316 stainless steel nuts welded to the inner surface of the head are used for suspending racks of surveillance coupons inside the vessel. Cracking was found on the machined top surface of the type 347 flange (Fig. 2) and in the weld connecting the nuts to the inside surface of the dissolver head. Reexamination in March 1979 showed no increase in cracking, but pitting was observed for the first time.

A type 316 stainless steel blind flange and thermowell assembly, also from the Wilsonville dissolver, was removed from service when



Fig. 2. Cracking Observed on Top Surface of Type 347 Stainless Steel Flange in the Wilsonville SRC Pilot Plant Dissolver. Residual stresses from machining combined with the corrosive environment to produce stress corrosion cracking.

cracks were observed on the flange plate. Metallographic examination revealed that the cracks penetrated a repair weld into the base metal, and more cracks were found in areas that had been ground after repair. The cross section of the flange in Fig. 3 shows the extent of cracking. Etching revealed that the cracking followed substructural boundaries in the weld and was transgranular in the base metal.

Severe cracking occurred in a type 316 stainless steel vent line on the piping between the preheater and dissolver at Wilsonville. The 1-in. sched-80 pipe was extensively cracked, with the transgranular cracks initiating from the inner surface (Fig. 4). Microprobe analysis showed some evidence of chlorides in the corrosion scale. Further details of these incidents are reported elsewhere.⁴⁻⁶

Stress corrosion cracking has also occurred at the Fort Lewis SRC pilot plant, primarily in the separator vessels downstream from the dissolver. These vessels are constructed of 2 1/4 Cr-1 Mo steel (high-pressure separator) or of C-1/2 Mo steel (intermediate-pressure separator and recycle condensate separator) and are clad with type 304

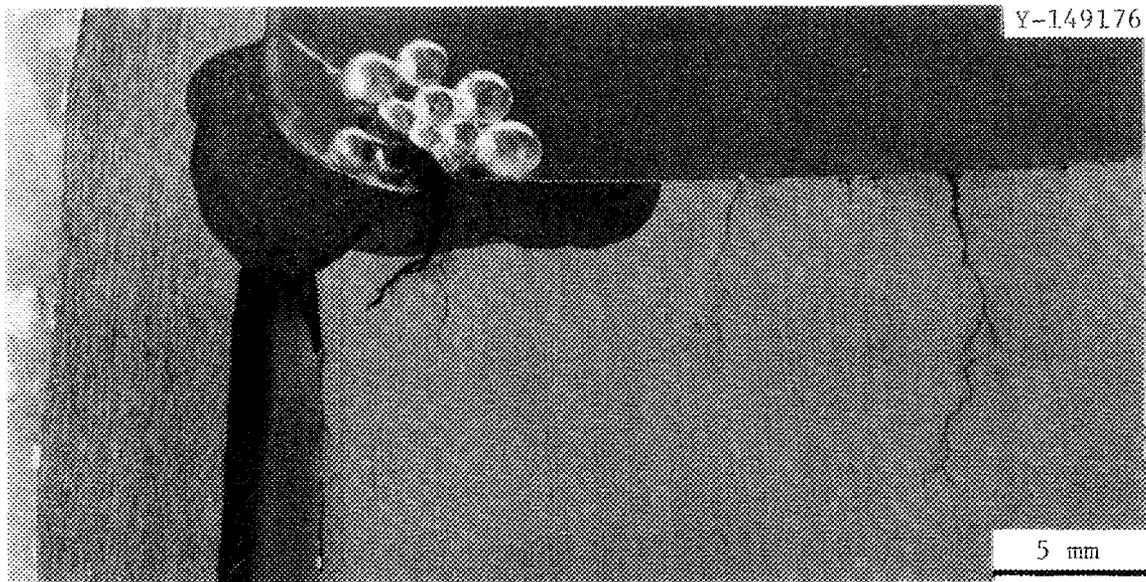


Fig. 3. Cross Section of Type 316 Stainless Steel Blind Flange and Thermowell Showing Cracks Through the Weld and in Areas Away from the Weld. Residual stresses resulted from the weld repair and subsequent grinding.

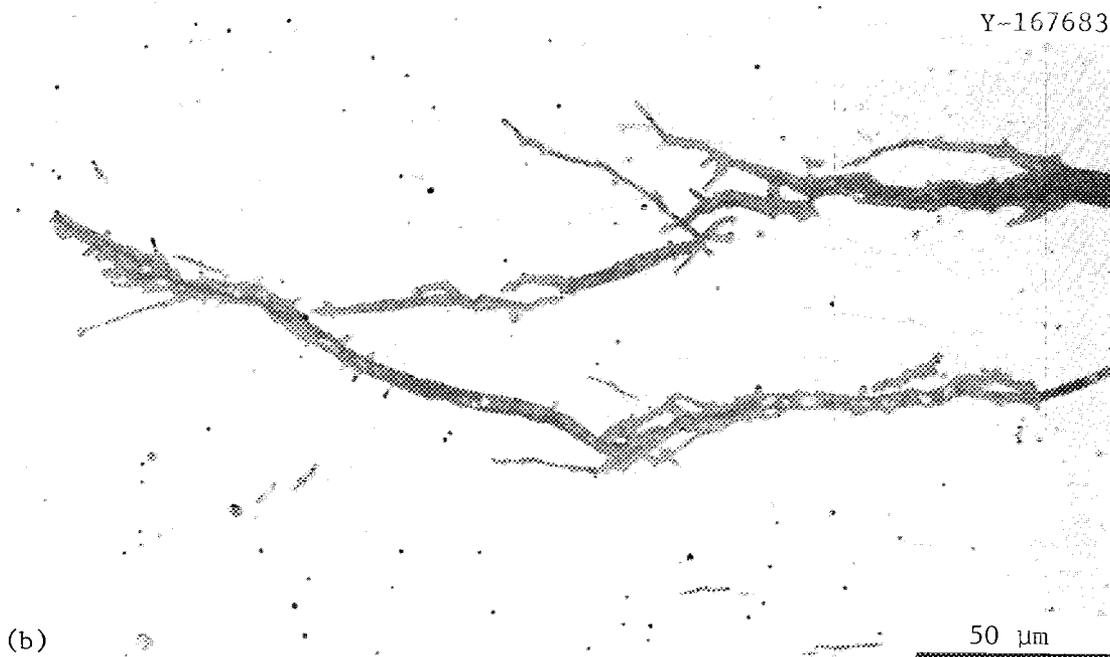
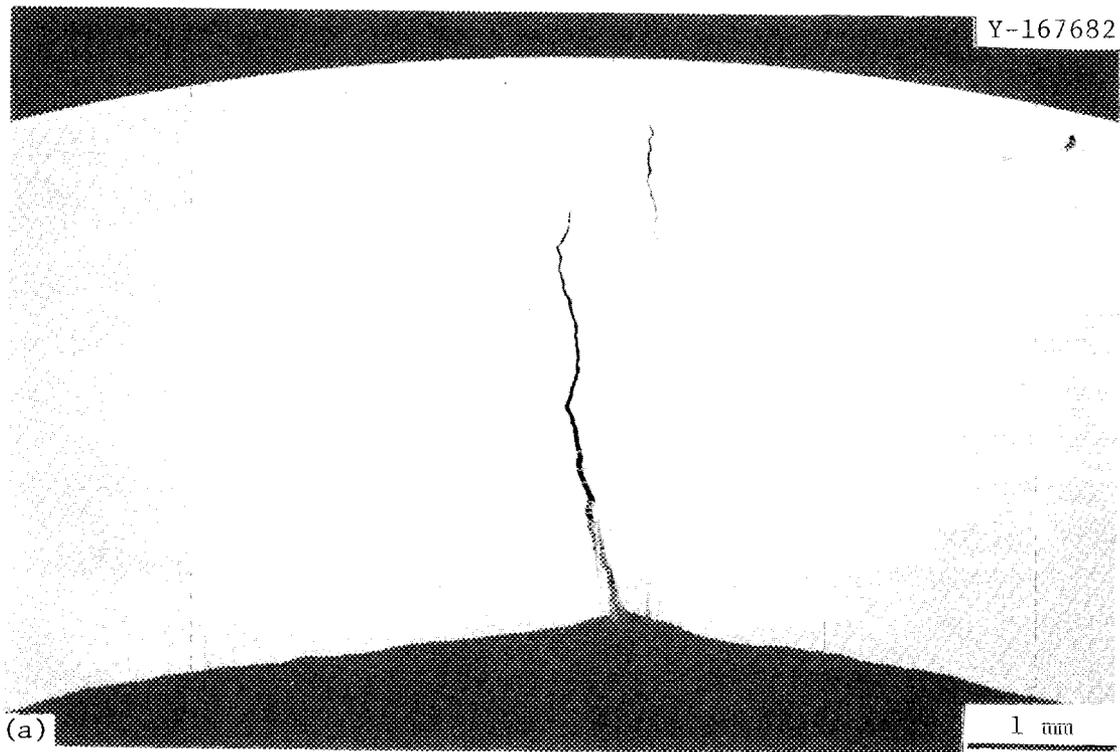


Fig. 4. One-inch Sched-80 Type 316 Stainless Steel Vent Line Off Piping Between Preheater and Dissolver at Wilsonville. (a) Cracks initiated at the inside diameter of the pipe and were observed on every sample cut. (b) Etching revealed the transgranular nature of the cracking.

stainless steel. Intergranular and transgranular stress corrosion cracks were found in stainless steel breather rings in the intermediate pressure separator and especially in the cladding on the vessel head. Cracking was also found in the cladding of the high-pressure separator and recycle condensate separator. The most severe cracking was in areas adjacent to welds, probably indicating localized sensitization of the cladding during fabrication. The cracking did not extend into the base metal. The majority of the cracks were intergranular and were attributed to polythionic acid attack, although some chloride-induced cracks were also found. Further details are available.⁷⁻⁹

Transgranular cracking attributed to chlorides was also found when a leak developed in the inlet nozzle of the high-pressure flash drum relief valve. The cracking was in the heat-affected zone (HAZ) of a weld between a type 316 stainless steel Grayloc hub and the type 316 stainless steel nozzle. All type 316 stainless steel components in the preheater-reaction areas were checked subsequently and cracking was found on one other inlet nozzle, that for the preheater inlet relief valve. Additional information is available.¹⁰

Isolated cases of stress corrosion cracking have occurred in other areas of both plants, such as in filter screens, valve seats, and other high stress areas. In general, the only area where cracking has shown a consistent pattern is the preheater-reaction-separation area, but even here, the cracking has been moderate and has not affected the plant operability significantly. However, because of concern for stress corrosion cracking in commercial-size plants, a DOE-sponsored program was initiated at ORNL to study the problem by means of plant and laboratory exposures of stressed specimens in coal liquefaction environments.

EXPERIMENTAL PROCEDURE

Racks of U-bend specimens were inserted into critical plant vessels where cracking had been observed or where a potential for cracking exists. Since much of the cracking occurred in areas adjacent to welds, specimens included both nonwelded and welded materials. The results are

intended to be correlated with laboratory tests designed to complement the pilot plant studies. The laboratory experiments are just under way and will be reported and correlated at a later date.

Most of the racks were located in the critical reaction-separation area. These vessels previously contained racks of flat surveillance coupons, and the U-bend racks were individually designed to be compatible in each location. The U-bend configuration was chosen because of its ease in fabrication and the size convenience for the plant operators. The stress caused by forming the U-bend shape is beyond that normally encountered in actual fabrication and thus is a very severe test. Test materials included ferritic stainless steels (e.g., 26-1 and 18-2), many austenitic stainless steels (including L grades and stabilized types), and various nickel-base alloys, such as Incoloy* 825, Inconels* 600 and 625 and Hastelloys† G and C-276.

Where possible, 12.7-mm-thick plates were used, but some were as thin as 1.6 to 6.4 mm. The 12.7-mm plates were welded together by multiple passes using the automatic gas tungsten-arc process with cold-wire filler additions. The joint configuration was a 75°-included-angle V-groove. All welds were radiographed to ensure a defect-free structure. Specimens were machined from the plates perpendicular to the weldment such that some specimens contained a weld cross section and others were nonwelded. The specimens were nominally 86 mm long, 13 mm wide, and 1.6 mm thick. Table 2 gives a complete list of specimen material, filler wire, and alloy composition. The thinner plates were not welded.

The specimens were weighed, bent into U-shapes, and attached to racks by nuts screwed onto bolts threaded on both ends. The racks were welded onto rods which are threaded for mounting in plant vessels. The configuration and dimensions varied from vessel to vessel, but some typical arrangements are shown in Figs. 5 and 6. Holes were machined in the rack to reduce the weight. Most of the racks were constructed of type 304L stainless steel; however, titanium and Hastelloy C-276 were used for

*Trademark of Huntington Alloys, Inc.

†Trademark of Cabot Corp.

Table 2. Composition of Alloys and Type of Filler Wire in U-Bend Specimens^a

Alloy	Heat	Type of Filler	Base Metal Composition, wt %									
			Cr	Ni	Fe	Nb	C	Si	Mn	P	S	Other
410	b	410	11.8	0.8	Bal	0.1	^c	0.2	0.9	^c	^c	
18-2	33178	26-1	18.00	0.56	Bal	1.83	0.016	0.70	0.25	0.025	0.001	Cu: 0.12, Nb: 0.33, Ti: 0.17
26-1	Unknown	26-1	26.30	0.12	Bal	1.00	0.0024	0.25	0.10	0.018	0.011	Ni: 0.018
304	b	308	19.2	8.3	Bal	0.32	^c	0.46	1.62	^c	^c	
304L	19971	308L	18.45	9.10	Bal	0.46	0.020	0.48	1.54	0.025	0.010	Cu: 0.44
310S	723988	310	24.79	19.40	Bal		0.050	0.68	1.88	0.25	0.001	
316	b	316	16.8	13.3	Bal	2.3	^c	0.64	1.64	^c	^c	
316L	10090	316 ELC	17.65	11.10	Bal	2.25	0.019	0.44	1.58	0.024	0.017	Cu: 0.46
317L	H7295	317L	18.74	13.95	Bal	3.22	0.019	0.59	1.62	0.020	0.018	
321	68089-1C	347	17.31	10.76	Bal		0.049	0.55	1.20	0.021	0.016	Ti: 0.37
347	68462-3C	347	18.39	10.93	Bal		0.043	0.60	1.43	0.025	0.019	Nb: 0.58, Ta: 0.02
332	Unknown	Inconel 82	20.26	31.22	Bal	0.15	0.015	0.44	1.00	0.025	0.001	Ti: 0.48
800H	7728A	Inconel 82	19.46	31.82	46.08		0.08	0.24	0.90		0.003	Cu: 0.54, Al: 0.43, Ti: 0.42
Carpenter 20Cb-3 ^d	19070	320	19.16	33.07	Bal	2.12	0.021	0.31	0.59	0.018	0.002	Cu: 3.23, Nb + Ta: 0.56
Incoloy 825	HH8464F		22.08	42.85	28.50	2.88	0.02	0.19	0.30		0.002	Cu: 2.24, Al: 0.06, Ti: 0.88
Inconel 600	HX1772	Inconel 82	14.83	75.91	8.45		0.06	0.21	0.29		0.007	Cu: 0.22
Inconel 625	HX4688AS	Inconel 82	22.21	61.25	3.20	9.10	0.02	0.18	0.05		0.005	Al: 0.16, Ta: 0.23, Nb + Ta: 3.57
Hastelloy G-3	8985-7- 7578		22.84	Bal	18.26	6.98	0.008	0.37	0.82	0.14	<0.005	Nb + Ta: 0.25, Co: 3.53, Cu: 1.84, W: 0.94
Hastelloy C-276	2760-7- 3709	C-276	16.07	Bal	6.18	15.82	0.004	0.04	0.47	0.012	0.002	V: 0.20, W: 3.49, Co: 1.95

^aVendor analysis except where noted.^bSemiquantitative analysis performed by ORNL Analytical Chemistry Division; heat number unknown.^cNot analyzed.^dTrademark of Carpenter Technology.

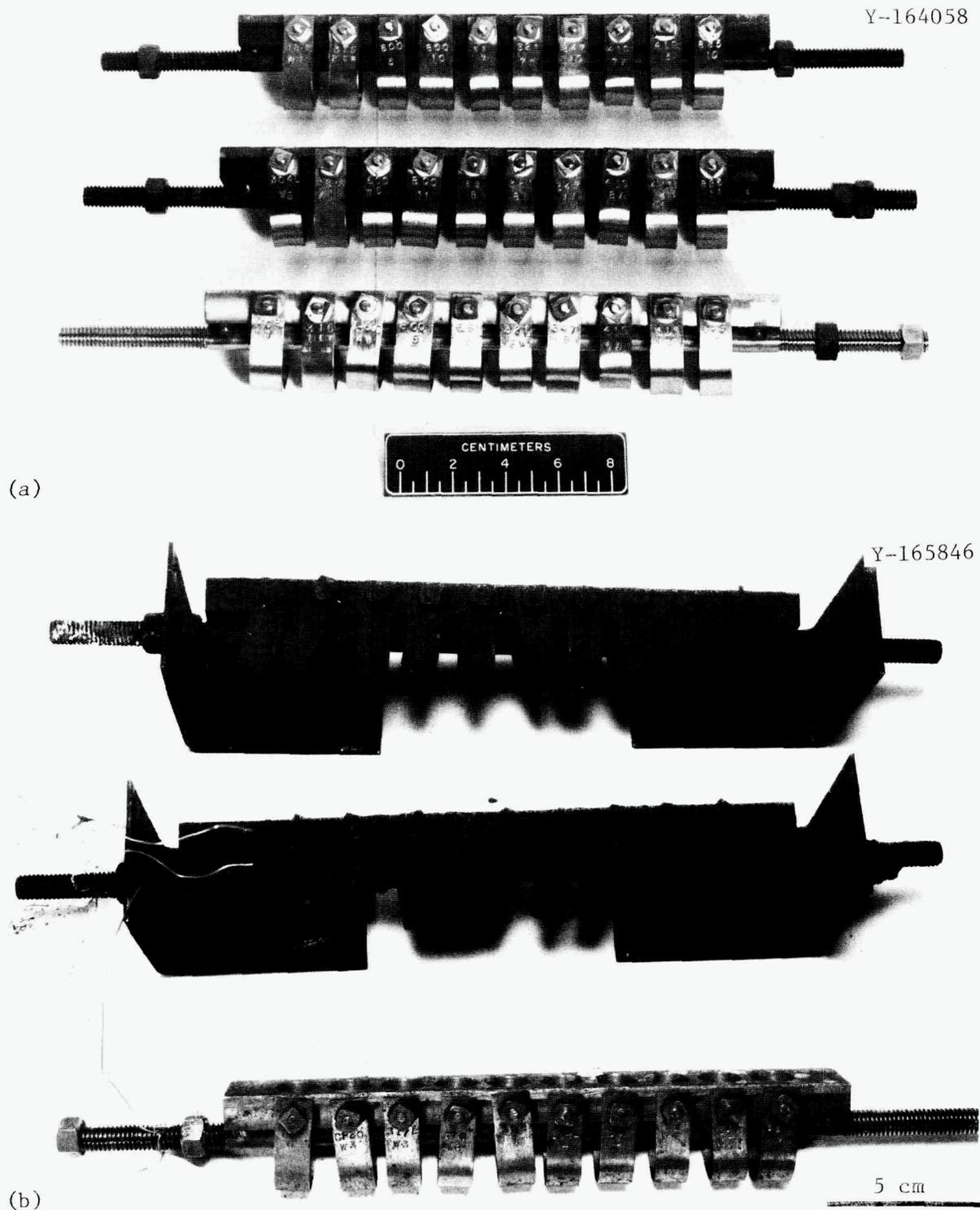
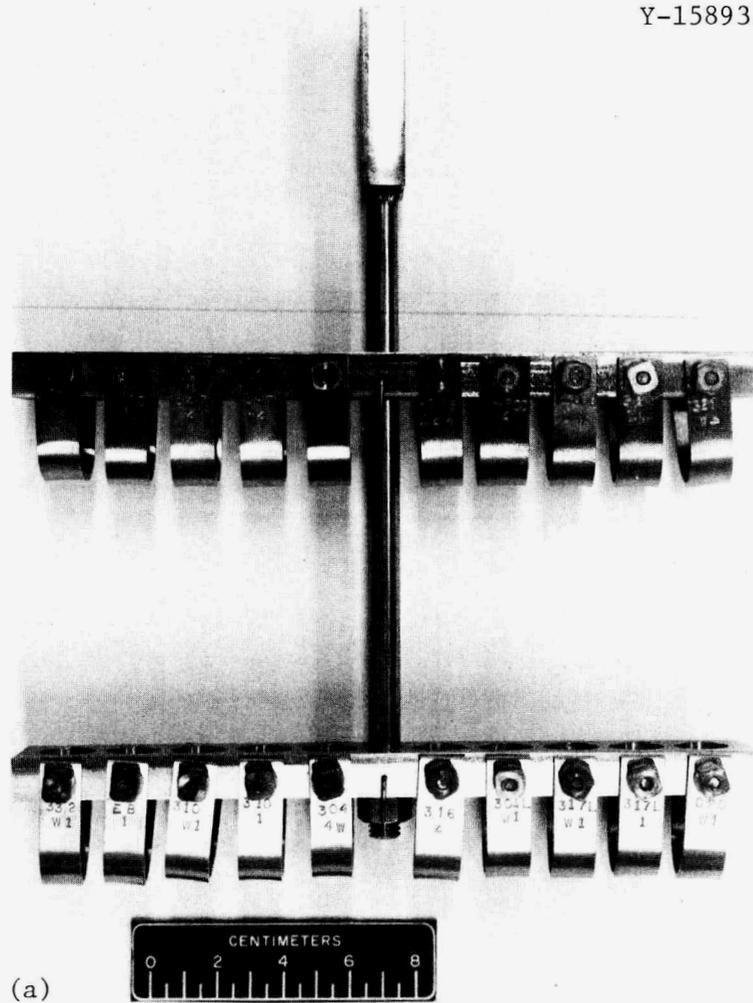
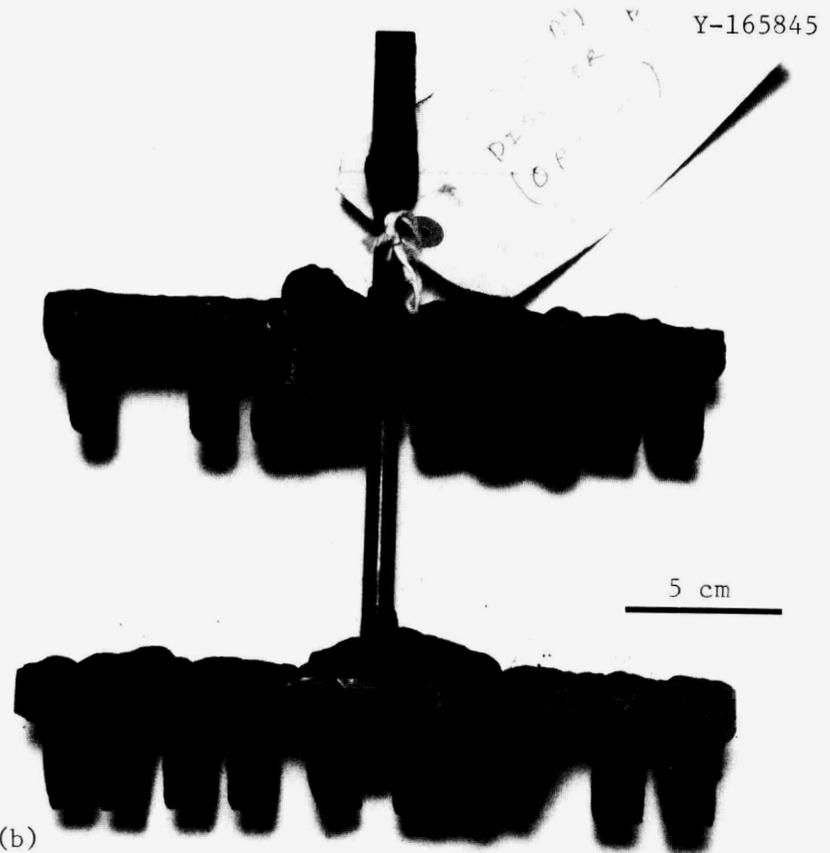


Fig. 5. Racks of Specimens Used for Mounting on Vessel Walls. The racks are mounted on brackets or screwed into nuts so that they are horizontal in the vessel. (a) Racks prior to exposure. (b) Similar specimens after exposure in the Fort Lewis separator vessels.

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(a)



(b)

Fig. 6. Rack Configuration Used for Suspending Specimens from Vessel Head. (a) Rack before insertion into the dissolver at the Fort Lewis SRC pilot plant. (b) Rack after exposure for 2800 h at operating temperature. Note the specimen missing from the left side of the top rack.

areas where corrosion was expected to be a problem. The mounting bolts were fabricated from alloy 800, titanium, and Hastelloy C-276 and solution annealed when necessary. The specimens, racks, and bolts were all mutually insulated by high-purity alumina washers. Specimens were not heat-treated, although future exposures will include sensitized specimens.

The time of exposure in each vessel varied for each exposure period, depending on plant operating conditions. Racks of specimens were inserted and removed at scheduled shutdowns; however, unscheduled operational interruptions may have occurred. The process conditions are difficult to characterize because the purpose of pilot plant operations is to study the effect of process variations. The conditions within any exposure period could have varied weekly if not daily, and thus it is virtually impossible to detail the exposure conditions other than by operating hours above a particular temperature.

After exposure, the specimens were returned to ORNL for analysis. They were examined visually for evidence of cracks, cleaned ultrasonically to remove corrosion deposits, and then reexamined. They were then removed from the racks and the amount of springback was measured. The specimens were weighed, and the corrosion rate was calculated based on an assumption of uniform removal of material. A dye-penetrant examination was performed on each specimen, and samples that gave any indication of attack were sectioned and examined by standard metallographic techniques.

Replacement racks were provided before a scheduled shutdown so that a new set of specimens could be installed in the vessel when the old ones were removed. The decision on materials to be tested in each location was made with the advice of plant personnel. Since the exposure period at Fort Lewis is shorter than that at Wilsonville, we have examined specimens from three periods at Fort Lewis while the specimens in similar vessels that were inserted in Wilsonville in March 1979 are still in place. This report summarizes the results of U-bend exposures in the dissolver and separator vessels only at the Fort Lewis SRC plant from January 1979 through June 1980. Specimens from the fractionation areas at both plants were also examined, and the results of these examinations are described.

Pilot plant exposures are still continuing at the SRC facilities, and U-bend samples have also been supplied to the Exxon Coal Liquefaction Pilot Plant and the H-Coal Pilot Plant. The results of these tests will be reported later.

RESULTS

Tables 3 through 8 list all specimens exposed at the Fort Lewis and Wilsonville SRC pilot plants that have been examined to date, along with the location, temperature, time of exposure, corrosion rates, and nature of attack (if any). Three exposure periods were completed in the Fort Lewis separator vessels (Tables 3-5), and a new set of specimens was inserted before each exposure. One test period was completed for the Fort Lewis dissolver and wash solvent column (Tables 6 and 7) and for the Wilsonville fractionation column (Table 8). The length of exposure was based on the number of operating hours at the temperatures indicated and thus varied with each vessel for each exposure period. Specimens were exposed to the vapor phases of the process streams.

The calculated corrosion rates for the dissolver and separator vessels (Tables 3-6) show that all the alloys tested (except Inconel 600) exhibited good to outstanding resistance to general corrosion in these environments. An alloy is considered to have good resistance if the corrosion rate is less than 0.5 mm/year.¹¹ Most of the specimens tested had corrosion rates of less than 0.1 mm/year and many corroded at a rate less than 0.001 mm/year. In general, the corrosion rates were highest for the high pressure separator and lowest for the recycle condensate separator. Since the temperature is higher in the former vessel, this observation is not surprising. During the same exposure period, specimens exposed in the dissolver had similar corrosion resistance to those exposed in the high pressure separator. The operating conditions of these vessels are very similar. The 26 Cr-1 Mo ferritic stainless steel exhibited the best corrosion resistance in the dissolver-separator environments. Inconel 600 clearly displayed the least corrosion resistance, as every specimen but one had disappeared from the test racks before they were removed for examination.

Table 3. Summary of U-Bend Specimens Exposed in the Fort Lewis High Pressure Separator at 330 to 430°C

Specimen Number ^a	Alloy	Exposure Period ^b	Corrosion Rate (mm/year)	Type of Attack ^c
410-4	410	3	0.45	P
410-1W	410	1	0.30	TC
410-8W	410	3	0.44	TC
18-2-1W	18-2	3	0.02	N
EB-1	26-1	1	<0.01	N
EB-8	26-1	3	<i>d</i>	N
EB-2W	26-1	2	<<0.01	IC ^e
304-1W	304	1	0.13	N
304L-2W	304L	2	0.03	GS
304L-6W	304L	3	0.13	IP
310-2W	310S	2	<<0.01	P
316-1	316	1	0.21	N
316-1W	316	1	0.22	N
321-2	321	2	0.03	GS
321-1W	321	1	0.12	N
321-8W	321	3	0.09	IP
347-1	347	1	0.11	N
347-11	347	3	0.06	IP
347-1W	347	1	0.09	N
347-5W	347	2	0.02	GS
332-1	332	2	0.02	P
I800-1	800H	1	0.12	N
I800-11	800H	3	0.03	S
I800-1W	800H	1	0.11	IC
CP20-2W	Carpenter 20Cb-3	2	0.03	IC
I825-11	Incoloy 825	3	0.04	N
600-1	Inconel 600	2	<i>f</i>	S
600-6W	Inconel 600	3	1.73	S,GS
625-1	Inconel 625	2	<<0.01	IC
C276-1W	Hastelloy C-276	2	<<0.01	TC,S

^a_W = welded specimen.

^b₁ = 736 h, 1/79-3/79; 2 = 2800 h, 3/79-10/79;
3 = 2707 h, 10/79-6/80.

^c_{GS} = general surface attack, IC = intergranularly cracked, IP = intergranular penetrations, N = no attack, P = pitted slightly, S = sulfidized, TC = transgranularly cracked.

^dSpecimen gained weight.

^eSuspected anomaly.

^fSpecimen destroyed completely by corrosion.

Table 4. Summary of U-Bend Specimens Exposed in the Fort Lewis Intermediate Pressure Separator at 280 to 380°C (536–716°F)

Specimen Number ^a	Alloy	Exposure Period ^b	Corrosion Rate		Type of Attack ^c
			(mm/year)	(mpy)	
410-3	410	3	0.06	2.4	GS
410-2W	410	1	0.05	2.0	P
410-7W	410	3	0.07	2.8	TC
18-2-4	18-2	2	<0.01	<0.1	N
18-2-2W	18-2	3	<0.01	<0.1	IC ^e
EB-2	26-1	1	<0.01	<0.1	N
EB-7	26-1	3	<i>d</i>	<i>d</i>	N
EB-3W	26-1	2	<i>d</i>	<i>d</i>	N
304-2W	304	1	0.03	1.2	N
304L-3W	304L	2	<<0.01	<<0.1	N
310-2A	310S	2	<i>f</i>	<i>f</i>	N
316-2	316	1	0.07	2.8	N
316-2W	316	1	0.05	2.0	N
316E-2W	316ELC	3	0.02	0.79	N
317L-2W	317L	2	<<0.01	<<0.1	N
321-3	321	2	<0.01	<0.1	N
321-2W	321	1	0.08	3.1	N
321-7W	321	3	0.04	1.6	N
347-2	347	1	0.05	2.0	N
347-10	347	3	0.01	0.39	N
347-2W	347	1	0.04	1.6	P
347-6W	347	2	<<0.01	<<0.1	N
332-2W	332	2	0.01	0.39	N
I800-2A	800H	1	0.05	2.0	N
I800-10	800H	3	0.02	0.79	N
I800-2W	800H	1	<0.01	<0.1	N
I825-10	Incoloy 825	3	<i>d</i>	<i>d</i>	N
600-2W	Inconel 600	2	<i>f</i>	<i>f</i>	S
600-5W	Inconel 600	3	1.48	58	S,GS
C276-2W	Hastelloy C276	2	<<0.01	<<0.1	N

^aW = welded specimen.

^b1 = 800 h, 1/79–3/79; 2 = 2800 h, 3/79–10/79; 3 = 2808 h, 10/79–6/80.

^cGS = general surface attack, IC = intergranularly cracked, N = no attack, P = pitted slightly, S = sulfidized, TC = transgranularly cracked.

^dSpecimen gained weight.

^eSuspected anomaly.

^fSpecimen destroyed completely by corrosion.

Table 5. Summary of U-Bend Specimens Exposed in the Fort Lewis Recycle Condensate Separator at Ambient Temperature to 180°C

Specimen Number ^a	Alloy	Exposure Period ^b	Corrosion Rate (mm/year) ^c	Type of Attack ^c
410-2	410	3	<0.01	N
410-3W	410	1	<0.01	TC
410-6W	410	3	<0.01	TC
18-2-6	18-2	2	<i>d</i>	N
EB-3	26-1	1	<0.01	N
EB-6B	26-1	3	<i>d</i>	N
EB-4W	26-1	2	<<0.01	N
304-1	304	2	<<0.01	N
304-3W	304	1	0.01	N
304L-4W	304L	2	<<0.01	N
310-3W	310S	2	<<0.01	N
316-3	316	1	<0.01	N
316-3W	316	1	<0.01	N
316E-1W	316ELC	3	<<0.01	N
317L-3W	317L	2	<<0.01	N
321-4	321	2	<<0.01	P
321-3W	321	1	<0.01	N
321-6WA	321	3	<i>d</i>	N
347-3	347	1	<0.01	N
347-9	347	3	<i>d</i>	N
347-3W	347	1	<0.01	N
347-7W	347	2	<<0.01	N
332-3	332	2	<<0.01	N
1800-3	800H	1	<0.01	N
1800-9	800H	3	<<0.01	N
1800-3W	800H	1	<0.01	N
CP20-3W	Carpenter 20Cb-3	2	<<0.01	N
1825-9B	Incoloy 825	3	<i>d</i>	N
600-4W	Inconel 600	3	<i>d</i>	N
G3-1A	Hastelloy G-3	3	<<0.01	N

^aW = welded specimen.

^b1 = 1175 h, 1/79-3/79; 2 = 4500 h, 3/79-10/79; 3 = 4340 h, 10/79-6/80.

^cN = no attack, P = pitted slightly, TC = transgranularly cracked.

^dSpecimen gained weight.

Table 6. Summary of U-Bend Specimens Exposed in the Fort Lewis Dissolver at 330 to 450°C for 2600 h (3/79-10/79)

Specimen Number ^a	Alloy	Corrosion Rate (mm/year)	Type of Attack ^b
410-4W	410	<i>c</i>	N
EB-1W	26-1	<i>c</i>	N
304-4W	304	0.03	P
304L-1W	304L	<i>c</i>	N
310-1	310S	<i>c</i>	N
310-1W	310S	<i>c</i>	N
316-4	316	0.05	N
316-4W	316	0.02	N
317L-1	317L	0.03	N
317L-1W	317L	0.04	N
321-1	321	0.06	N
321-4W	321	0.08	N
347-4	347	0.04	N
347-4W	347	<<0.01	N
332-1W	332	0.30	N
I800-4	800H	0.29	TC, S
I800-4W	800H	0.35	TC, S
CP20-1W	Carpenter 20Cb-3	0.09	N
600-1W	Inconel 600	<i>d</i>	S
625-1W	Inconel 625	0.03	TC, S

^aW = welded specimen.

^bN = no attack, P = pitted slightly, S = sulfidized, TC = transgranularly cracked.

^cSpecimen gained weight.

^dSpecimen destroyed completely by corrosion.

Table 7. Summary of U-Bend Specimens Exposed in
the Top of the Fort Lewis Wash Solvent Column
at 180-280°C for 1334 h (10/79-1/80)

Specimen Number ^a	Alloy	Corrosion Rate (mm/year)	Type of Attack ^b
EB-5W	26-1	2.39	GS
316-6W	316	1.05	GS
316L-1W	316L	0.87	GS
317L-6W	317L	0.91	P
321-6WB	321	0.91	GS
CP20-4W	Carpenter 20Cb-3	0.34	GS
1825-9A	Incoloy 835	0.54	GS
625-3W	Inconel 625	0.33	GS
G3-1B	Hastelloy G-3	0.39	GS

^aW = welded specimen.

^bGS = general surface attack, P = pitted.

Table 8. Summary of U-Bend Specimens Exposed for 5341 h in the Wilsonville Fractionation Column (6/79-4/80)

Specimen Number	Alloy	Temperature Range (°C)	Corrosion Rate (mm/year)	Type of Attack ^a
EB-4	26-1	190-220	<0.02	N
EB-5	26-1	290-310	<i>b</i>	N
EB-6A	26-1	220-260	<i>c</i>	GS, P
304L-1	304L	190-220	<0.02	N
304L-3	304L	220-260	0.48	TC, P
304L-4	304L	290-310	<i>b</i>	N
310-2B	310S	190-220	<0.02	N
310-3	310S	220-260	0.38	TC
310-5	310S	290-310	<0.01	N
316-5	316	190-220	<0.02	N
316-6	316	220-260	0.47	GS
316-7	316	290-310	<i>b</i>	N
317L-2	317L	190-220	<0.02	N
317L-4	317L	220-260	0.55	GS
317L-5	317L	290-310	<i>b</i>	N
321-5	321	190-220	<0.02	N
321-7	321	220-260	0.38	TC, P
321-8	321	290-310	<0.01	TC, P
347-5	347	190-220	<0.02	N
347-7	347	220-260	0.43	TC, P
347-8	347	290-310	<0.01	TC, P
I800-2B	800H	190-220	<0.02	N
I800-7	800H	220-260	0.26	TC
I800-8	800H	290-310	0.30	S
CP20-1	Carpenter 20Cb-3	190-220	<0.02	N
CP20-3	Carpenter 20Cb-3	290-310	<0.01	N
CP20-4	Carpenter 20Cb-3	220-260	0.01	IP
600-2	Inconel 600	190-220	<0.02	N
600-4	Inconel 600	220-260	0.01	IP
600-5	Inconel 600	290-310	<0.01	IP

^aGS = general surface attack, IP = intergranular penetrations, N = no attack, P = pitted, TC = transgranularly cracked.

^bSpecimen gained weight.

^cSpecimen destroyed completely by corrosion.

Visual examination of the first set of specimens exposed in the separator vessels revealed no evidence of cracking. However, a dye-penetrant check showed indications on a few specimens and these were examined metallographically. All the type 410 stainless steel welded samples were pitted or cracked. Some of the cracks initiated in the weld metal and penetrated through the HAZ and others originated in the base metal. The type 410 specimen that was exposed in the high pressure separator is shown in Fig. 7. The cracking is a classic case of stress corrosion, initiating at a surface pit and then branching transgranularly through the metal. Intergranular cracking was found on one alloy 800H specimen. No austenitic stainless steel specimens were cracked, although a few showed pitting near the weld fusion line. All other specimens were unattacked.

Visual and metallographic examination of the second set of specimens tested in the dissolver and separator vessels revealed no attack on specimens exposed in the recycle condensate separator or the intermediate pressure separator. Several specimens exposed in the high pressure separator were cracked or corroded. The Hastelloy C-276 specimen (Fig. 8) showed transgranular sulfidation, and Inconel 625 (Fig. 9) and Carpenter 20Cb-3 showed intergranular cracks. The Inconel 625 specimen was non-welded; however, the cracks in the other specimens occurred in the welds. Weld area cracking was also observed in two specimens exposed in the dissolver, alloy 800H (Fig. 10) and Inconel 625 (Fig. 11). In general, the high-nickel alloys performed poorly because of sulfidation as expected.

The third round of testing in the Fort Lewis separator vessels yielded similar results to the previous exposures. The amount of attack (both general corrosion and cracking) was greater in the high pressure separator than in the other two vessels; however, except for the high-nickel alloys, the corrosion rate was still within acceptable ranges. The 26 Cr-1 Mo displayed the best resistance to corrosion and cracking. Most of the type 410 specimens exhibited transgranular cracking and/or heavy pitting. The high-nickel alloys were again sulfidized, but the attack was not as severe as was noted previously for the specimens from the dissolver vessel. Three austenitic stainless steel specimens in the high pressure separator showed small intergranular penetrations after 2707 h: types 304L, 321, and 347 (Fig. 12).

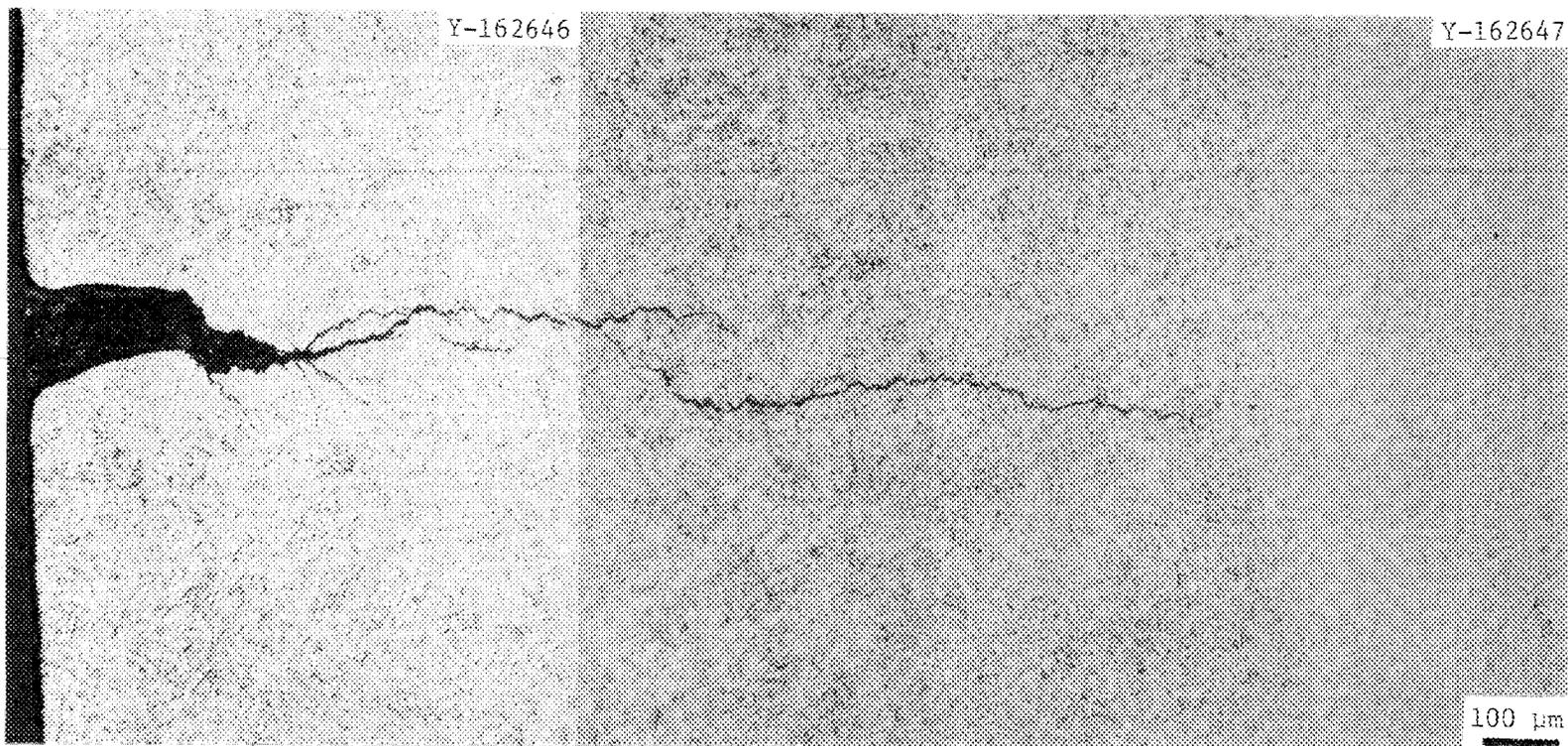


Fig. 7. Crack in Type 410 Stainless Steel U-Bend Specimen Exposed for 736 Operating Hours in the High Pressure Separator at Fort Lewis. The crack initiated at the surface and penetrated through the base metal and heat-affected zone before stopping at the weld.

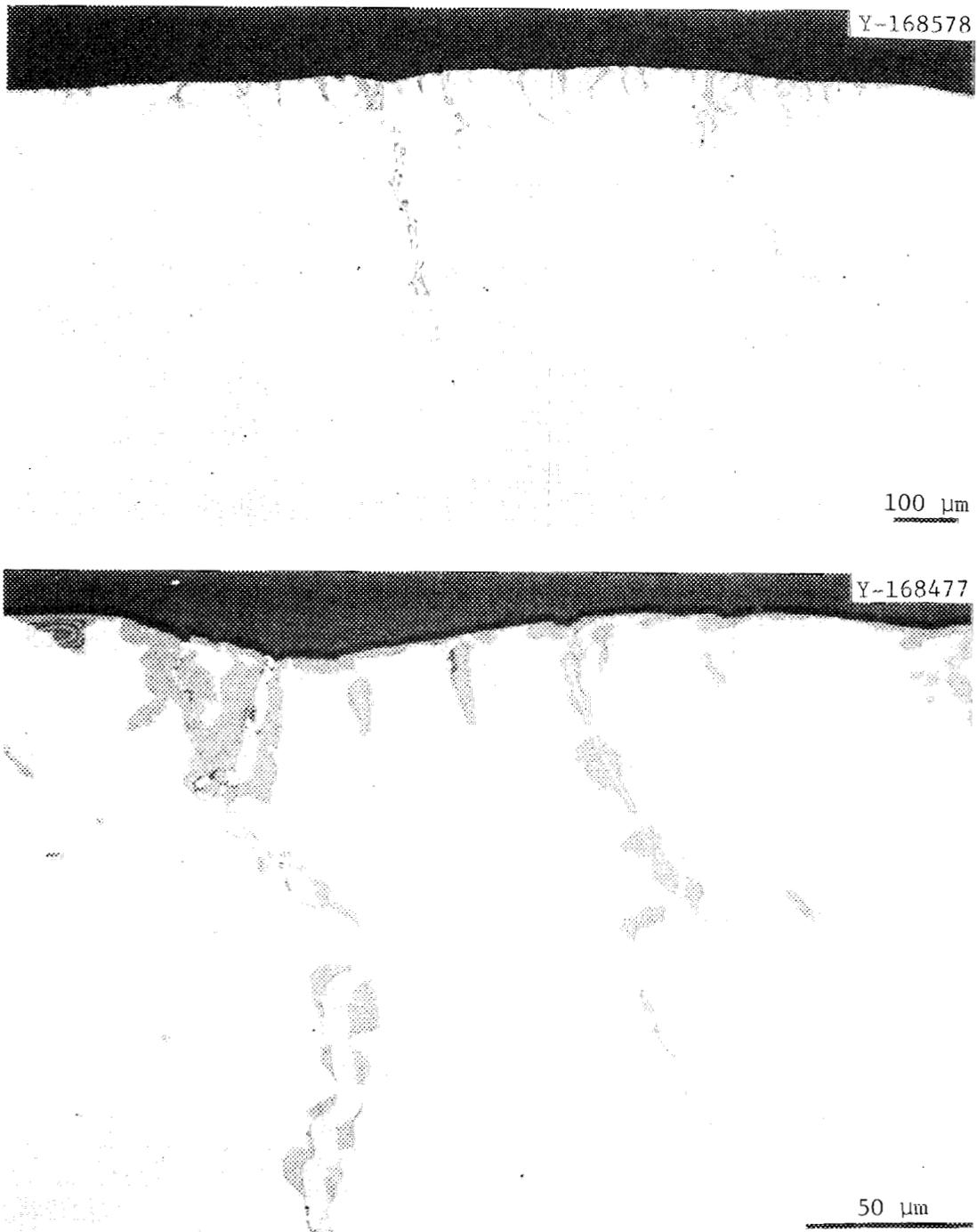


Fig. 8. Hastelloy C-276 U-bend Specimen After Exposure in the Fort Lewis SRC Pilot Plant High Pressure Separator (2800 h).

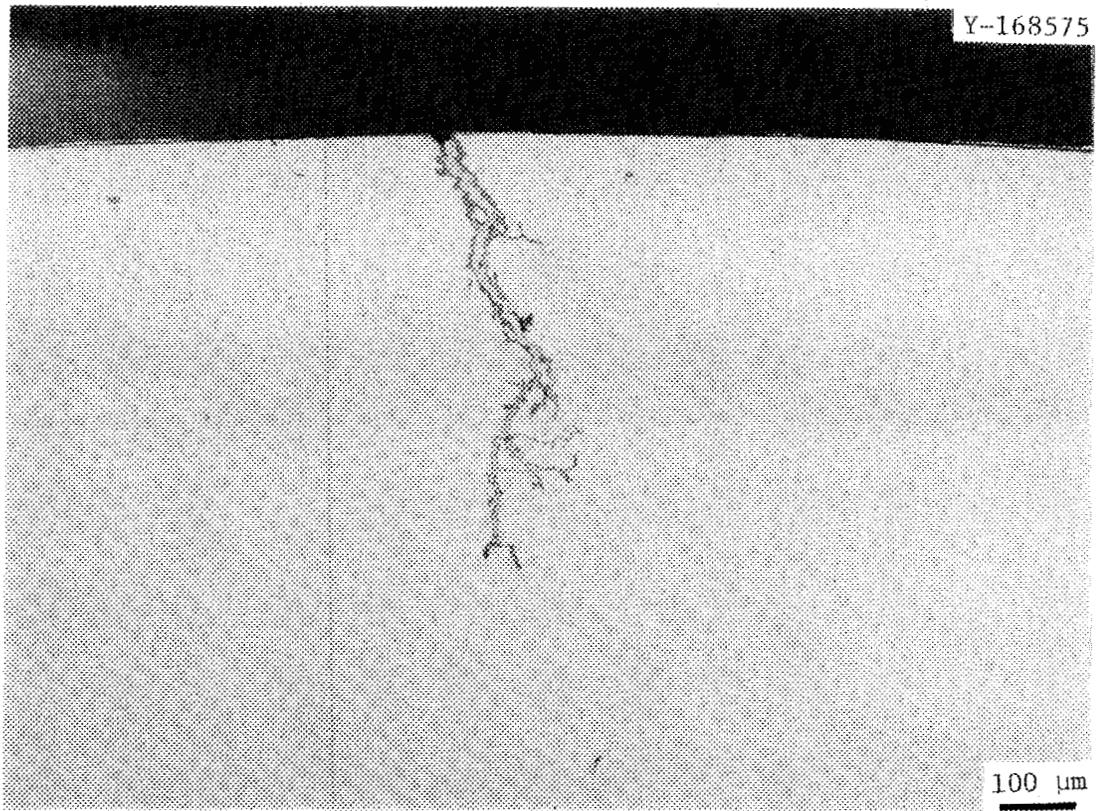


Fig. 9. Inconel 625 U-Bend Specimen Cracked After Exposure in the High Pressure Separator at Fort Lewis (2800 h at Temperature).

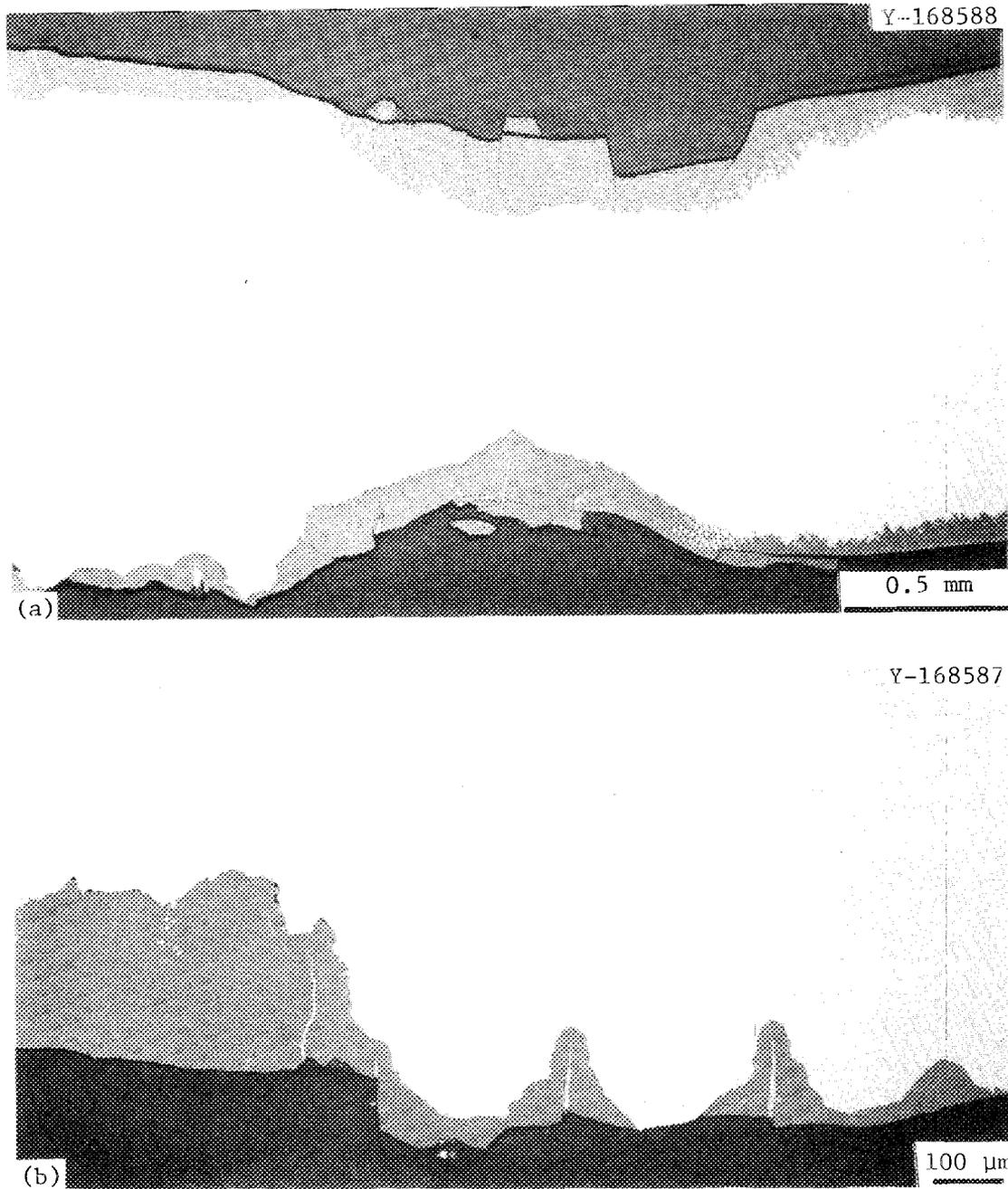


Fig. 10. Alloy 800H Specimen Was Extensively Attacked, Particularly Near Weld. This U-bend had been exposed in the Fort Lewis dissolver for 2600 h at operating temperature. (a) Cracking and sulfidation led to thinning of weld. (b) Sulfidation attack occurred from the cracks to more fully penetrate the alloy.

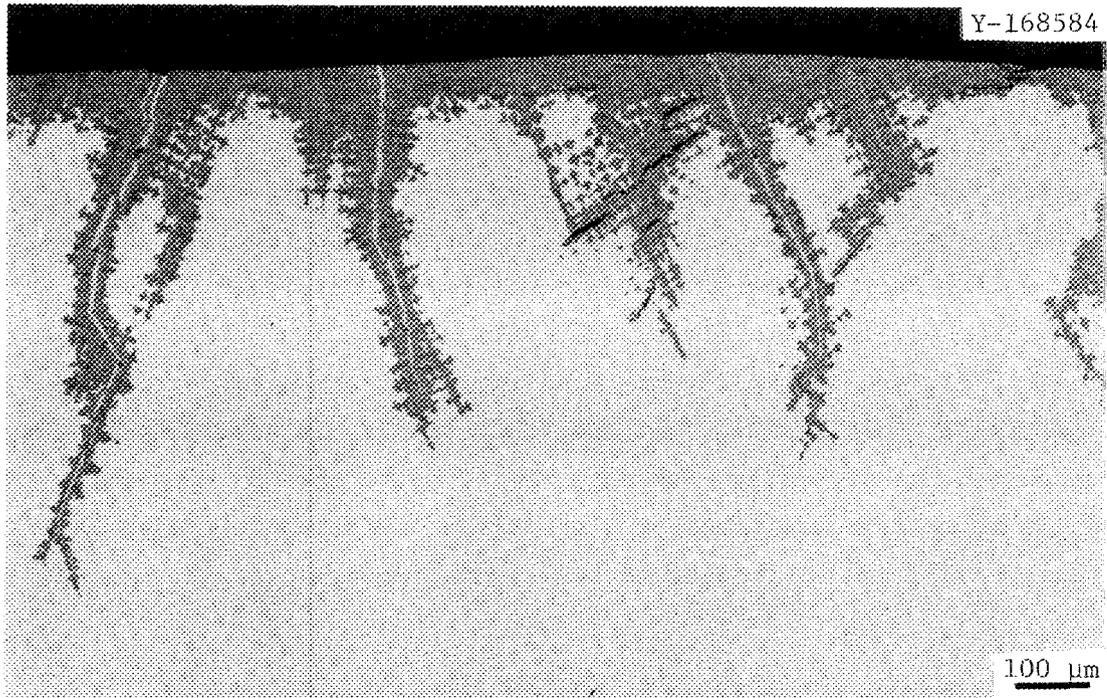


Fig. 11. Sulfidation Attack and Cracking of Inconel 625. This U-bend specimen was exposed in the Fort Lewis dissolver for 2600 h at operating temperature. Note the dendritic nature of the penetrations, indicating that the attack is in the weld.

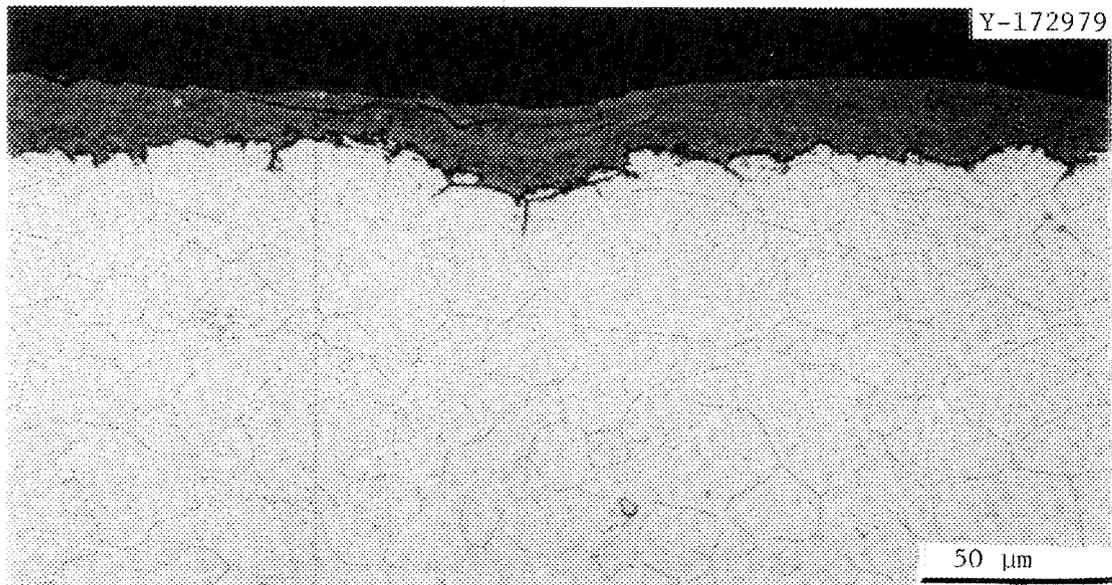


Fig. 12. Intergranular Penetrations Were Observed in Austenitic Stainless Steels After 2707 h of Exposure in the Fort Lewis High Pressure Separator. Section is from a type 347 stainless steel U-bend.

The performance of these materials differed considerably in the Fort Lewis wash solvent column (Table 7) and the Wilsonville fractionation column (Table 8). The 26 Cr-1 Mo had the highest corrosion rate (>2.3 mm/year), and the high-nickel alloys showed the best resistance to attack; however, intergranular attack was found on alloy 800H (Fig. 13) and Inconel 600 (Fig. 14). The fractionation area environment contains considerably less sulfur and is generally at a lower temperature than the area around the dissolver, thus minimizing sulfidation. The austenitic stainless steels performed poorly in this area, with corrosion rates of around 1 mm/year at Fort Lewis. The lower corrosion rates at Wilsonville are due to the use of sodium carbonate additions to control corrosion. These additions were necessitated by a greatly accelerated corrosion that began in 1978 and is thought to be associated with organic compounds and



Fig. 13. Intergranular Cracking Occurred on Alloy 800H U-Bend Specimen Exposed for 5341 h in the Middle of the Fractionation Column at Wilsonville.

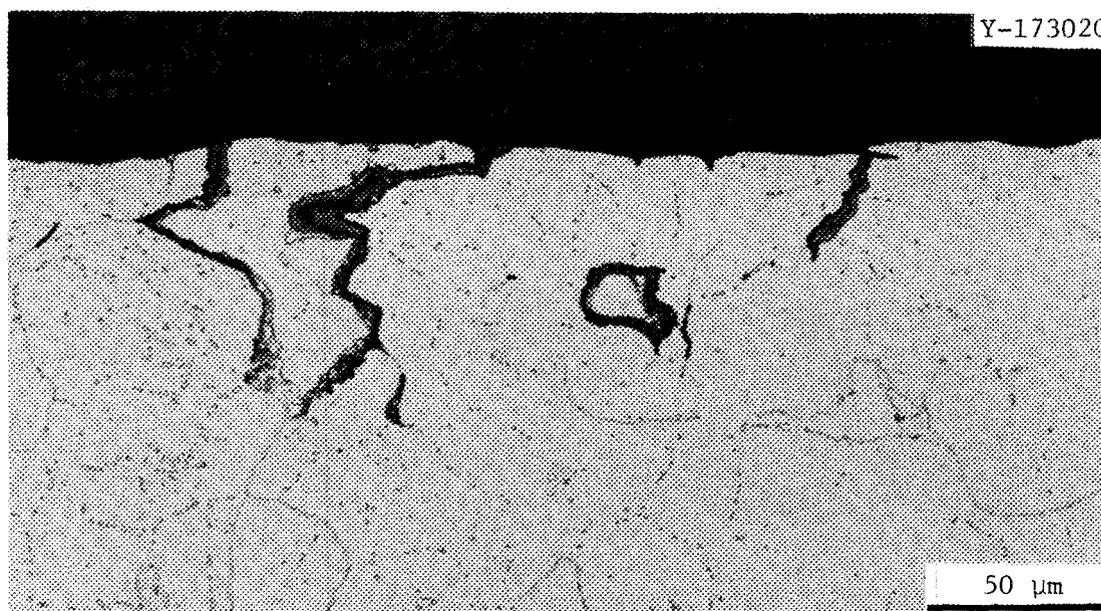


Fig. 14. Inconel 600 U-Bend Specimen Was Intergranularly Attacked After 5341 h Exposure in Wilsonville Fractionation Column Near the Middle Manway.

chloride.¹² The high corrosion rates have been endemic to this area at Fort Lewis since startup in 1974.^{3,7} Most of the attack on the austenitic stainless steel U-bends at Fort Lewis was a general surface attack with some pitting. More pitting was found on the Wilsonville specimens, with a considerable amount of transgranular cracking on types 304L, 310S, 321, and 347 (Figs. 15-18). The cracking network on these specimens is very similar to the chloride-induced cracking of the type 316 vent line (Fig. 4). Extensive intergranular cracking of the type 304 cladding had been observed in this vessel at the same time the corrosion rate increased.¹²

DISCUSSION

The view generally held by process developers is that the coal liquefaction streams after dissolution are very similar to crude oil refinery streams (except for the presence of solids), and that, therefore, the materials problems should be amenable to current oil industry practices. Most of the cracking observed in petroleum refineries is intergranular and is usually attributed to polythionic acid attack.¹³⁻¹⁸

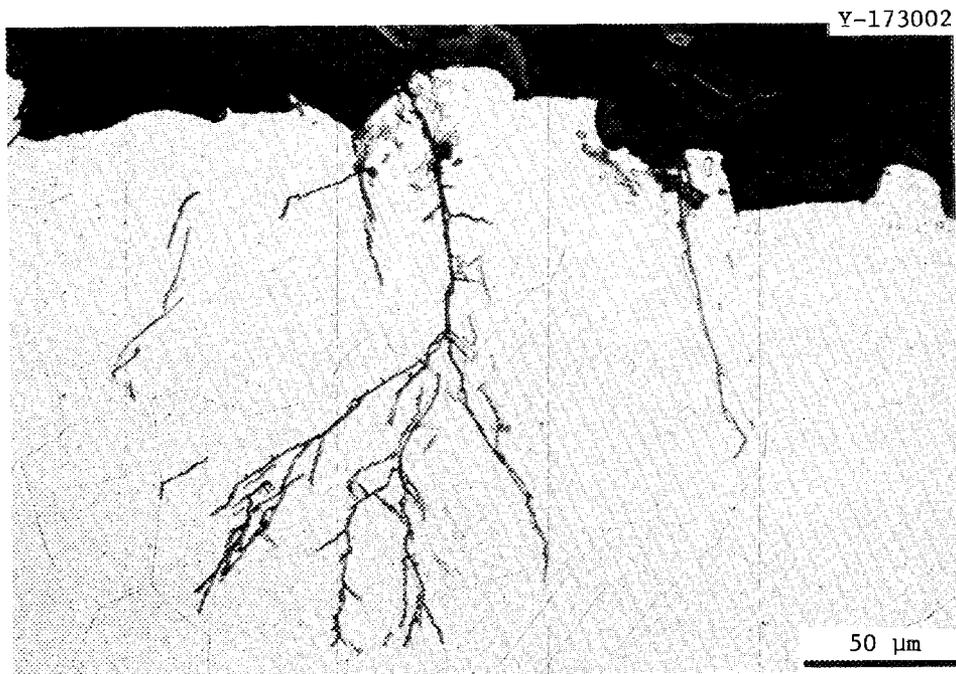


Fig. 15. Transgranular Cracking on Type 304L Stainless Steel U-Bend Exposed for 5341 h in the Middle of the Wilsonville Fractionation Column.

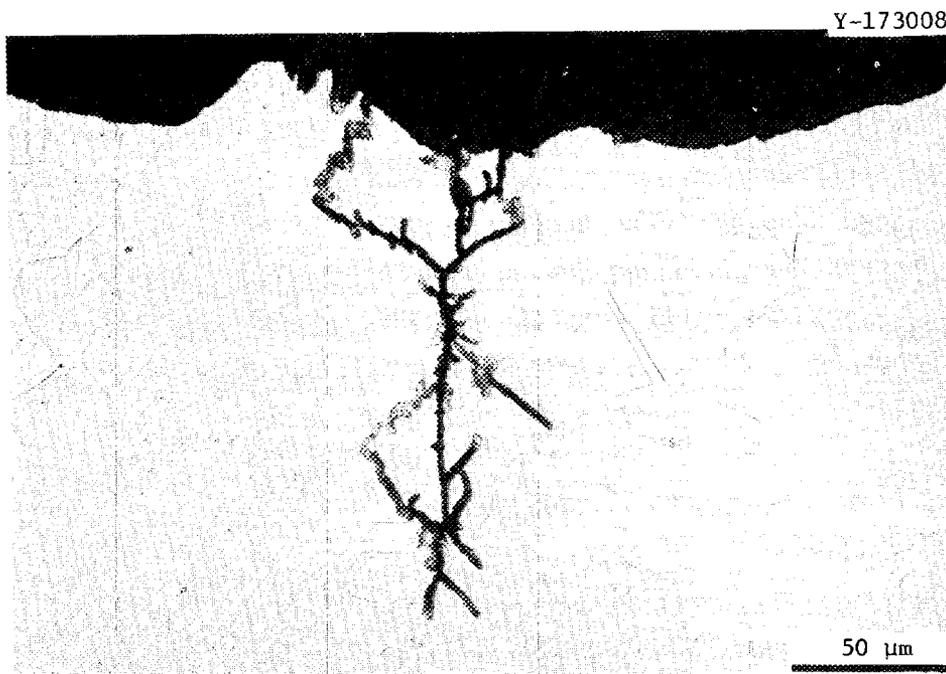


Fig. 16. Type 310S Stainless Steel U-Bend Specimen Displayed Transgranular Cracks After 5341 h Exposure in the Middle of the Fractionation Column at the Wilsonville SRC Pilot Plant.

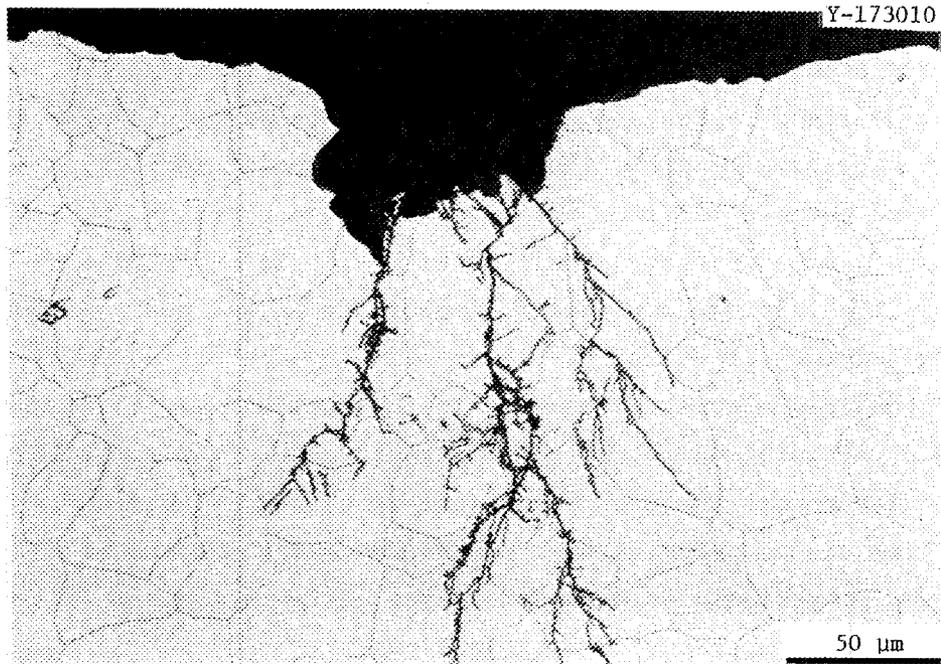


Fig. 17. Transgranular Cracking of Type 321 Stainless Steel U-Bend Specimen Exposed for 5341 h in the Middle of the Wilsonville Fractionation Column.

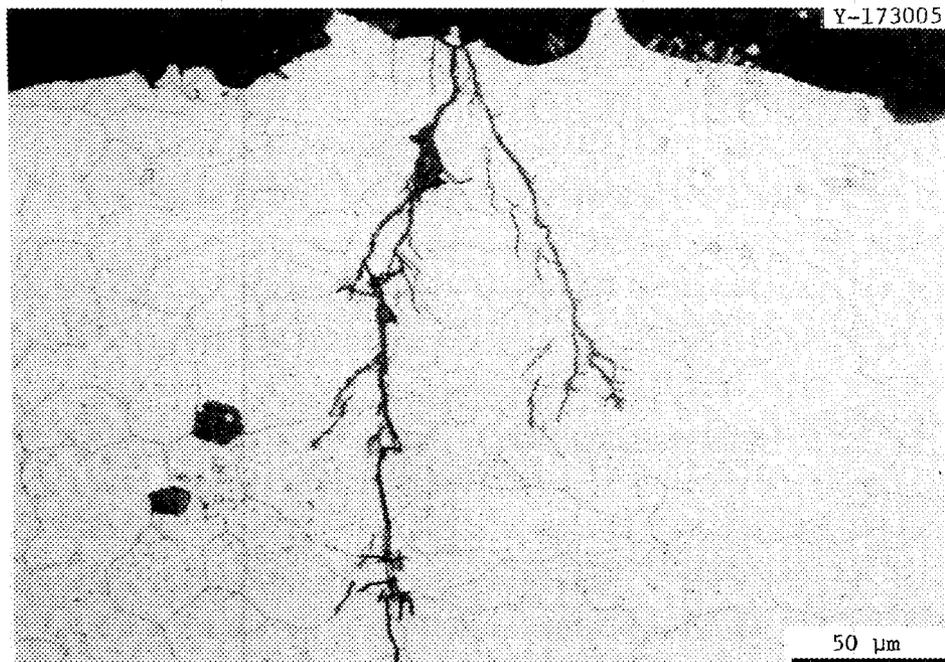


Fig. 18. Type 347 Stainless Steel U-Bend Specimen Exposed in the Middle of the Wilsonville Fractionation Column for 5341 h Showed Transgranular Stress Corrosion Cracking.

Generally, polythionic acid stress corrosion cracking occurs in austenitic stainless steels that are sensitized; that is, they have a chromium-depleted region adjacent to grain boundaries at which chromium carbides have precipitated. The formation of polythionic acid requires an interaction between sulfide, moisture, and oxygen, which are present in coal liquefaction systems as well as in refineries. Sulfide is present in the coal liquefaction environment, especially in and near the reaction area, in the form of H_2S or as a corrosion product on the metal surface. The moisture may already be present in the coal but may also result from washing or steaming the vessel during shutdowns. Oxygen is obtained from the air that enters whenever the vessel is opened. Thus, while the high temperature of operation is what causes the sensitization of the stainless steel, the polythionic acid does not form until the vessel is exposed to air at ambient temperature during shutdown.

Polythionic acid attack is controlled in refineries by materials selection and/or precautions taken during shutdowns. The use of the stabilized grades of stainless steels (types 321 and 347) is recommended where polythionic acid formation may be expected. These steels contain alloying additions that form carbides of greater stability than chromium carbide and thus maintain corrosion protection. The other method used for controlling polythionic acid stress cracking is specified in NACE Standard RP-01-70, Recommended Practice for "Protection of Austenitic Stainless Steel in Refineries Against Stress Corrosion Cracking by Use of Neutralizing Solutions During Shut Down." The use of soda ash or ammonia solutions to wash down the stainless steel surfaces has been shown to control the problem; however, this procedure is seldom used for low-alloy steel vessels with stainless steel overlay because it is difficult and expensive.^{14,16} Consequently, neutralizing solutions have not been used generally during shutdowns at coal liquefaction plants, although polythionic acid cracking has been found. It is expected that future generation vessels will be made of a low-alloy steel overlaid with annealed type 347 stainless steel and thus no problems with polythionic acid cracking are anticipated.^{16,19-21}

While refinery experience deals very satisfactorily with the intergranular cracking caused by the formation of polythionic acids, there

is no similar information dealing with the transgranular cracking caused by chlorides. Blom¹³ suggests that polythionic acid solutions containing chlorides will cause transgranular stress corrosion cracking and Brophy¹⁴ mentions transgranular chloride cracking; however, most of the emphasis is placed on polythionic acid attack. It is suspected that the lack of emphasis on chloride attack is due to its infrequency in refineries since crude oils contain relatively little chloride. On the other hand, the chloride content of the coal feed may be as much as 0.29% (by weight), considerably more than the less than 50 ppm generally encountered in desalted crudes. Thus, while chloride cracking is not a common problem in refineries, it may pose a very significant problem in coal liquefaction systems.

Experience at the Wilsonville plant has shown that chloride-induced transgranular cracking of austenitic stainless steels has occurred. The presence of chlorides has also been associated with the severe general corrosion problem in the fractionation area.¹² The austenitic stainless steel U-bends examined from this area clearly display the type of transgranular cracking usually associated with chlorides. Chloride cracking of type 316 stainless steel was observed in the EDS demonstration unit in areas where condensation of water occurred.²² Although most of the cracking observed at Ft. Lewis was intergranular and attributed to polythionic acid attack, some chloride stress cracking was also identified.

Examination of the U-bend specimens exposed at the Ft. Lewis SRC pilot plant has yielded some information on the types of alloys that may be suitable for certain critical plant locations. In vessels at elevated temperature with a sulfur-rich environment, the nickel-base alloys, as a class, tend to be unsuitable since they are susceptible to sulfidation. Other materials that performed poorly in the dissolver-separator area were alloys 800 and type 410 stainless steel, both of which were cracked in more than one specimen. The austenitic stainless steels performed well in this high-temperature, sulfur-rich environment, although they exhibited some tendency toward pitting. Since these tests were relatively short term, it is difficult to assess the long-term effects of the localized attack. It is possible that these pits may have eventually acted as initiation sites for cracking.

In the lower temperature and sulfur environment of the wash solvent column, the nickel-base alloys exhibited superior corrosion and cracking resistance. The 26 Cr-1 Mo alloy should not be considered for this application. Also, the austenitic stainless steels are not satisfactory in this environment. The corrosion problems in the fractionation areas at both SRC plants are under intensive investigation and are more fully explored elsewhere.^{7,12,23}

Generally, we found that the presence of a weld, even including the possibly sensitized material in the HAZ, did not affect the cracking resistance of the alloy. Those alloys that were attacked, such as alloy 800H and type 410 stainless steel, showed cracking in the welds and in areas that were not welded. We also observed, however, that where a specimen was attacked in both regions that, in general, the attack was more severe in the welded area.

Since the U-bend exposures at Fort Lewis were basically short-term tests under uncontrolled conditions, laboratory tests have been initiated at ORNL to complement the plant exposures. These tests will include exposures in sulfur acids containing chloride additions, and autoclave exposures of U-bend specimens and constant extension rate tests in simulated environments. The latter test is much more severe than the U-bend test because it continually produces cracks or defects in the normally protective passive film. In these controlled conditions, further studies will be made to assess the cracking potential, particularly of the austenitic stainless steels. Exposure of U-bends in the pilot plants is also continuing. Furthermore, it is intended that an attempt be made to incorporate overlaid materials into the testing program, although we recognize the difficulty in approximating the stress state and overlay configuration with the U-bend test. The results of these continuing examinations will be reported later.

The experience with cracking in refineries is very relevant to what has occurred in the SRC pilot plants, especially with regard to polythionic acid attack; however, enough differences exist between these technologies to warrant careful examination. In particular, while many of the organic compounds in coal liquefaction process streams are also

present in crudes, there are larger variations in relative amounts of these compounds in the former, especially with regard to chloride content. Austenitic stainless steels are notoriously susceptible to chloride stress cracking. Therefore, the selection of 300-series stainless steels to counter polythionic acid environments could lead to inadequate resistance to chloride attack in coal-derived liquids. It must be noted also that it is risky to assign environmental blame on the basis of crack morphology. It has been observed in other systems, most notably pure water, that chlorides can be associated with intergranular cracking.²⁴⁻²⁸ Thus, cracks that have been dismissed as due to polythionic acid and thus easily avoidable may in reality pose a more serious problem. These unknowns necessitate complete documentation of materials experience in operating plants and research into factors that may control stress corrosion behavior in coal liquefaction systems. In particular, further investigation of the effect of chloride levels in coal and the combined attack of polythionic acid and chlorides on stainless steels is necessary before any austenitic stainless steels can be safely recommended for long-term use in the critical preheater-dissolver-separation regions.

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