Report of Operating Experiences in Coal Conversion Plants
Failure Mode - Corrosion

William A. Willard
John H. Smith

Failure Prevention Information Center
Institute for Materials Research
National Bureau of Standards
Washington, D.C. 20234

Summary Report
December 1977

This work was sponsored by
Department of Energy
Washington, D.C. 20545
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U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, Secretary
Dr. Sidney Harman, Under Secretary
Jordan J. Baruch, Assistant Secretary for Science and Technology
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Acting Director
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Certain trade names and company products are identified in order to adequately describe the effect of the coal conversion processes on materials and components. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the products are necessarily the best available for the purpose.
Foreword

The NBS Failure Prevention Information Center is a centralized computer-based system for gathering, critically evaluating, and disseminating information about operating experiences, material evaluation, and component failures in coal conversion plants. Information sources include the coal conversion process development units and pilot plants and ERDA sponsored laboratories conducting diagnostic failure analyses. The objective of this program is to establish a reliable data base of component malfunctions and materials performance which will be useful in extending the lifetimes and reliability of plant components and which will help minimize the possibility of plant shutdowns.

An information gathering network has been developed for the reporting of all significant operating incidents and component failures to the NBS Failure Prevention Information Center. The reports are reviewed and evaluated for completeness and accuracy. A detailed technical summary is prepared of each incident and the information is indexed by process, date, material, failure mode, component category, and information source. The technical summary and indexed information are stored on a computer database management system (Computer Corporation of America Model 204) for ease of retrieval and analysis. This information is analyzed to identify significant problem areas, to determine corrective actions for solving component problems, and to conduct failure mode analyses for coal conversion plants.

It is especially intended that the information in the data base reach plant designers and plant operators. However, access to the Information Center is available to all interested parties. The information in the data base is being disseminated in several ways. All direct inquiries to the Information Center are answered. Reports of failures and summaries of diagnostic failure analysis reports are published in the ERDA Newsletter, Materials and Components in Fossil Energy Applications(1), which is published bimonthly. This report is an example of the type of summary reports of operating experiences that will be prepared and issued by the Failure Prevention Information Center with emphasis on failure mode, materials of construction and plant components.

(1) The Newsletter is published by Battelle, Columbus Labs, 505 King Avenue, Columbus, Ohio 43201. Editor: R. Schorr.
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<td>Carbonate</td>
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<td>Removes sulfur oxides from power plant stack gases</td>
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<td>Clean Coke</td>
<td>Clean Coke Process Development Unit</td>
<td>Converts low grade, high sulfur coke to low sulfur, low ash coke and liquid and gaseous fuels</td>
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<td>COED</td>
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<td>CPC</td>
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<td>Uses the high pressure, fluidized-bed combustion of coal to produce a hot gas stream for driving a gas turbine generator</td>
<td>Combustion Power Company, Inc. Menlo Park, CA</td>
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<td>Exxon</td>
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<td>Converts coal to medium BTU fuel gas</td>
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<td>Research Projects</td>
<td>Grand Forks Energy Research Center, Grand Forks, ND</td>
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<td>Converts coal to high BTU gas</td>
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Abstract

This report consists of a group of summaries of operating experiences at coal conversion pilot plants, materials evaluation reports, and diagnostic failure analysis reports dealing with the problem of corrosion in the coal conversion industry. Reports received up to June, 1977 are included. The corrosion summaries are categorized into subgroups of specific types of corrosion: aqueous, carburization, metal dusting, oxidation, pitting, sulfidation and material evaluation. Within each subgroup of corrosion the summaries are grouped by component type (piping, pumps, valves, etc.) in alphabetical order. Each component category is sorted by coal conversion process (such as CO₂, Bygas, Synthene, etc.) in alphabetical order and each process is sorted by the date of the report from the earliest to the latest.

If further information is needed on any summary in this report, please contact the Failure Prevention Information Center and refer to the Information Number (I.N.) located at the top of each summary.

Acknowledgement

The authors wish to thank Mrs. Shirley Harrington, ADP Operations Division, Department of Commerce and Mrs. Dianna Mills and Miss Judy Bolden, Metallurgy Division, National Bureau of Standards for their help in preparing this report.
AQUEOUS
FAILURE ANALYSIS REPORT, 10/31/74, BY A. MAC NAB, C. F. BRAUN AND CO.
FAILURE OF CARBON STEEL CONDENSER TUBING

SUMMARY

PITTING AND NONUNIFORM CORROSION OF CARBON STEEL PIPE IN HEAT EXCHANGER.
ATTACK IS FROM INSIDE OF TUBING INDICATING AN AGGRESSIVE ENVIRONMENT IN
PROCESS RAW GAS CONDENSATE. PIPE IS 3/4" OD X 0.965" W.T. CARBON STEEL IN
SERVICE FOR 6 MONTHS GASIFICATION OUT OF THREE YEARS USE. ENVIRONMENT:
PROCESS CONDENSATE IS HIGHLY ACID. AMMONIA ADDED FOR pH CONTROL.
METAL LOSS: 53 MILS MAX, 33 MILS AVG.

* * * * *

FOSSIL ENERGY ADVISORY REPORT NO. 12, 3/26/75.
HYGAS PILOT PLANT-CONDENSER TUBING-RAW GAS QUENCH COOLER

SUMMARY

MATERIAL: CARBON STEEL
FLUID: CHI SANITARY DISTRICT CANAL WATER OUTSIDE. PROCESS CONDENSATE, NH3
NEUTRALIZED INSIDE.
DISCUSSION: UNIT IN SERVICE 3-1/2 YRS., OPERATED 1-1/2 YRS UNDER GASIFICATION
CONDITIONS. CORRODED. AVG. METAL LOSS 10-20 MILS. MOST CORROSION PROBABLY
OCCURRED DURING SHORT UNINHIBITED PERIODS OF OPERATION SIGNIFYING THAT THE
PROCESS FLUID IS HIGHLY "AGGRESSIVE" TO CARBON STEEL.
RECOMMENDATION: AVOID EVEN SHORT PERIODS OF ACIDIC CONDITION OR USE MORE
RESISTANT MATERIAL.
SAMPLE HISTORY: 2 SECTIONS OF 310 S.S. 1/4" OD SCHEDULE 40 TUBING WERE RECEIVED FOR A METALLOGRAPHIC EXAMINATION. ONE SAMPLE WAS EXPOSED TO 1400-1700 F TEMP. IN A FLUIDIZED BED COMBUSTOR. EXPOSURE TIME NOT STATED. THE SECOND SAMPLE WAS REPRESENTATIVE OF PRIOR FLUIDIZED-BED TREATED MATERIAL.

EXAMINATION: TUBES HAD BEEN FABRICATED WITH A SEAM WELD WHICH IS BAD FROM A CORROSION AND CYCLIC STRESS VIEWPOINT. THE FLUIDIZED BED EXPOSURE ERASED THE DENDRITIC MICROSTRUCTURE IN THE WELD BY RECRYSTALLIZATION. A NEW PHASE THAT APPEARS TO BE SIGMA HAS FORMED IN THE WELD AND BASE METAL. THE SEAM WELD NOTCHES WERE SERIOUSLY AGGRAVATED BY THE FLUIDIZED BED EXPOSURE.

COMMENTS: CONSIDERATION SHOULD BE GIVEN TO THE USE OF SEAMLESS TUBING TO AVOID SEAM WELDS AND THE PROBABILITY OF SUBSEQUENT CORROSION FATIGUE FAILURES. THE PRESENCE OF SIGMA PHASE HAS BEEN REPORTED TO RESULT IN A LOWER DUCTILITY AND SEVERE LOSS OF NOTCH IMPACT STRENGTH IN 25CR-20NI STEELS.

SEE I.N. 371, 372, 373, 374, 375, 376 FOR BACKGROUND INFORMATION.

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OPERATING CONDITIONS—FLUIDIZED BED EXCHANGER TUBES

SUMMARY

BACKGROUND INFORMATION PERTAINING TO PIPE SECTIONS METALLOGRAPHICALLY EXAMINED BY R. GRAY, ORNL. SEE I.N. 367 FOR REPORT.

MATERIAL: 310 S.S. SCHEDULE 40

OPERATING CONDITIONS: COOLED WITH UNTREATED TAP WATER. EXIT WATER TEMP. WAS 200 F. EXPOSURE TIME—160 HRS. 10-100 CYCLES FROM AN UNFIR ED TO A FIRED CONDITION.

COMMENT: THE BED TEMPERATURE WAS RAISED TO 2000 F TO INITIATE COMBUSTION OF THE COAL BEFORE THE COOLING WATER WAS PASSED THROUGH THE TUBES. EACH EXPOSURE TO 2000 F (10-100 CYCLES) LASTED ABOUT 10 MINS. IT IS NOT UNLIKELY THAT THE CORROSION IN THE EXAMINED TUBES IS THE RESULT OF THIS 2000 F EXPOSURE.
I.N. PROCESS SEARCH CRITERIA
80 MISC. PIPING

PAPER BY R. OSMAK, EXXON, AT 1974 AIChE SYMPOSIUM ON AMMONIA PLANTS
HEAT EXCHANGER PROBLEMS IN A 1500 ST/SD AMMONIA PLANT

SUMMARY

1. CO2 REMOVAL SYSTEM REBOILER TUBES: AN APPARENT ATTEMPT TO STRESS RELIEVE 304 S.S. U-BENDS LED TO CARBIDE PRECIPITATION AT THE GRAIN BOUNDARIES AND HENCE TO TUBE FAILURES FROM INTERCRYSTALLINE CRACKING. THIS OCCURRED IN ONLY ONE BUNDLE. OTHER BUNDLES INCLUDING REPLACEMENT O.K.

2. WASTE HEAT BOILER TUBES: DEPOSITION OF BOILER FEED WATER IMPURITIES IN THE U-BENDS OF THE VERTICAL WATER TUBE WASTE HEAT BOILER LED TO TUBE FAILURES FROM OVERHEATING AND CORROSION. C-1/2MO STEEL TUBES. 1500 PSIG BOILER FEEDWATER (600°F) FLOWS THROUGH TUBES. HOT (1655°F) SECONDARY REFORMER EFFLUENT FLOWS ON THE SHELLSIDE. CURE-CLEAN UP WATER SUPPLY. INSULATE OUTSIDE SURFACE OF EACH U-BEND.

3. SYNGAS COMPRESSOR INTERCOOLER BUNDLES: COOLING WATER FLOWING ON THE SHELLSIDE OF CARBON STEEL COOLER TUBES AND HAVING RELATIVELY POOR WATER TREATMENT CAUSED RAPID TUBE CORROSION FAILURES, FORCING FREQUENT BUNDLE REPLACEMENT. CURE-CLEAN UP WATER SUPPLY. REDESIGN BUNDLES. COAT TUBES WITH PHENOLIC EPOXY.

4. A BIMETALLIC SHELL DESIGN IN A FIXED TUBESHEET EXCHANGER INTENDED TO MINIMIZE "AXIAL" THERMAL EXPANSION DIFFERENCES LED TO CRACKING AT THE SHELL TO TUBESHELL JUNCTION DUE TO THE "RADIAL" THERMAL EXPANSION DIFFERENCE. TUBES ARE C-1/2 MO, THE SHELL IS PART CARBON STEEL AND PART 304 S.S.. CRACKS WERE GROUND OUT AND WELDED WITH INCONEL 182 FILLER WIRE. REPLACEMENT WILL BE REDESIGNED AND ALL PARTS MADE FROM C-1/2 MO STEEL.

5. LOW TEMPERATURE SHIFT FEED COOLER: AN INLET DISTRIBUTOR CHANGE IN A CROSSFLOW EXCHANGER LED TO RAPID TUBE FAILURES FROM FLOW INDUCED VIBRATION CURE-REDESIGN USING 9 TUBE SUPPORT BAFFLES VS THE ORIGINAL 4.

* * *
CARBURIZATION
GOOD PROCESS DESCRIPTION OF THE WESTINGHOUSE COAL GASIFICATION SYSTEM.

FAILURES:
1. NOZZLE PLATE AND NOZZLE EXPERIENCED A SEVERE FORM OF HIGH TEMP. CORROSION BY CARBURIZATION AND OXIDATION DUE TO EXCESSIVE TEMP. AND ALTERNATING REDUCING-OXIDIZING ENVIRONMENTS.
2. MELTING OF SHROUD MATERIAL WAS RESULT OF LOCAL OVERHEATING CAUSED BY CRACKING OF THE NOZZLE PLATE-PILOT INLET HOUSING WELD, PROBABLE MALFUNCTION OF SEVERAL NOZZLES, AND ACCUMULATION OF HEAVY CARBON DEPOSITS AHEAD OF THE BURNERS.
3. CRACKING IN THE SYNTHESIS GAS GENERATOR WAS RESULT OF EMBRITTLEMENT DUE TO EXCESSIVE CARBIDE PRECIPITATION AND THERMAL STRESSES EXPERIENCED DURING CYCLING.

RECOMMENDATIONS:
1. REDESIGN BURNER SECTION OF THE GENERATOR SO THAT HIGH TEMP. OF OPERATION CAN BE AVOIDED.
2. USE INCONEL 601 WHICH HAS GOOD RESISTANCE TO CARBURIZATION AND OXIDATION.
3. EXPLORE THE USE OF REFRACTORY MATERIALS AND WATER COOLING OF BURNER COMPONENTS TO PREVENT FUTURE GAS GENERATOR FAILURES.
I.N. PROCESS SEARCH CRITERIA
17 CO2 PIPING

LETTER REPORT, 2/16/70, FROM J. LETERLE, CONOCO.
HIGH TEMPERATURE CORROSION OF PARTS FROM CO2 PILOT PLANT

SUMMARY

REACTOR VESSEL-310 S.S. THERMOWELLS-HASTELLOY X AND INCONEL. ALL METALS
SEVERELY CORRODED. TEMP. RANGE 1400-2000F. ATMOSPHERE, N2, CO2, CN, H20,
SO2, AND H2S.

CONCLUSIONS
1. IT APPEARS THAT SULFUR HAS REACTED WITH ALL THREE METALS TO FORM A LOW
   MELTING POINT EUTECTIC OF SULFUR, CHROMIUM, NICKEL, AND IRON.
2. THE 310 S.S. HAS BEEN HIGHLY CARBURIZED.
3. IF THE ENVIRONMENT THAT CAUSED THIS CORROSION IS TYPICAL THEN CAREFUL
   CONSIDERATION MUST BE GIVEN TO ALLOY SELECTIONS.

* * * *

I.N. PROCESS SEARCH CRITERIA
124 CO2 PIPING

INTEROFFICE REPORT FROM H. CROWDER TO F. J. RADD, CONOCO, 8/25/70.
METALLURGICAL STUDY OF REACTOR AND GASIFIER VESSELS FROM THE LIGNITE
GASIFICATION PRE-PILOT PLANT, CONOSIL

SUMMARY

THE BENCH SCALE LIGNITE GASIFICATION PLANT, OPERATED AT LIBRARY, PA HAS
EXPERIENCED HIGH TEMPERATURE CORROSION AND EMbrittLeMENT PROBLEMS.
THIS REPORT SUMMARIZES WORK ON THE FOLLOWING PARTS:
1. GASIFIER VESSEL
2. REGENERATOR VESSEL
3. REGENERATOR PRE-HEAT COIL
4. REGENERATOR THERMOWELL

CONCLUSIONS: ALL FOUR OF THE PIECES HAVE EXPERIENCED ONE OR MORE OF THE
FOLLOWINGS PROBLEMS: HIGH TEMPERATURE SULFUR CORROSION, EMBRITTLMENT
FROM CARBURIZING, EMBRITTLMENT FROM NITRIDING.
THESE PROBLEMS COVER THE TEMPERATURE RANGE OF 950-1900 F.
MET. ANALYSIS OF CORRODED INCOLOY 800 TUBE FROM A RECYCLE GAS HEATER.

SUMMARY

THE TUBE HAD A VERY COMPLEX HISTORY. NOT REPRESENTATIVE OF NORMAL OPERATING CONDITIONS.

CONCLUSIONS

1. THE PIPE HAS BEEN DEGRADED BY A COMBINATION OF CARBURIZATION, SULFIDATION, AND OXIDATION.
2. CARBURIZATION IN DEPTH PRECEDED THE OTHER REACTIONS AND ESTABLISHED A CR-RICH PHASE AT THE GRAIN BOUNDARIES.
3. INCOLOY 800 DOES NOT APPEAR TO HAVE ADEQUATE RESISTANCE TO OXIDATION IN THE ENVIRONMENT FOR THIS APPLICATION. A HIGHER CR ALLOY WHICH CAN FORM A MORE PROTECTIVE OXIDE FILM TO RETARD THE DIFFUSION OF CARBON AND SULFUR TO THE UNDERLYING METAL MAY GIVE BETTER PERFORMANCE. ADDITIONS OF AL, TI, AND SI ALSO WOULD BE HELPFUL.

EVALUATION OF HEATER TUBES FROM RAPID CITY CONSOL

SUMMARY

FIVE HEATER TUBE SAMPLES WERE SUBMITTED. THEY HAD BEEN INSPECTED BY MAGNETIC INSPECTION WHICH INDICATED SOME DAMAGE.
A MICROSTRUCTURAL EVALUATION WAS REQUESTED TO CORRELATE ACTUAL DAMAGE WITH MAGNETIC INSPECTION RESULTS.
ALONIZED 304 S.S. TUBE WAS GOOD. THE FOUR SAMPLES OF INCOLOY 800 TUBING CONTAINED VARYING DEGREES OF CORROSION AND STRUCTURAL DAMAGE. THE DAMAGE WAS < 12.2% OF THE MINIMUM MEASURED WALL THICKNESS.
SUMMARY

FAILURE: A 9-FT 9-IN SECTION OF OLD MATERIAL IN THE "A" PASS WAS REPLACED DUE TO THINNING AND A SMALL PIN HOLE.

SERVICE LIFE: 581 HRS SINCE REBUILD.

PRIOR REPAIRS: COIL WAS REBUILT REPLACING ALL THIN SECTIONS IN DEC. 1974 WITH ALONIZED RA-330.

ENVIRONMENT: Temp. 1000-1500°F. INERT GAS (83-87% N2, 6-12% CO2, 0.2-9.6% O2) AND RECYCLE GAS (80% N2, 17% CO2, 3% CO, CARBONYL SULFIDE 50 PPM).

MATERIALS OF CONSTRUCTION: INCOLOY 800 WITH SECTIONS OF ALONIZED RA-330.

CAUSE OF FAILURE: PROBABLY DUE TO CARBURIZATION AND SULFIDATION.

ACTION: DIAGNOSTIC ANALYSIS TO BE PERFORMED BY CONOCO.

* * * * *

SUMMARY

FAILURE: FURNACE TUBES HAVE EXPERIENCED FREQUENT TROUBLE FROM THINNING.

THINNING ORIGINATES FROM INSIDE OF TUBES.

SERVICE LIFE: UNKNOWN

ENVIRONMENT: 900°F. 3-4% CO, 26% CO2, 70% N2, 20-75 PPM COS.

MATERIAL: 321 S.S. 1-5/8" OD X 0.109" W.T.

RESULTS

1. INITIALLY GET CARBURIZATION, NITRIDING, AND SOME SULFIDATION THEN GET OXIDATION.

2. SULFUR ATTACK OCCURS, BUT NOT ENOUGH TO BE SIGNIFICANT AT THESE TEMPERATURE.

3. MAJOR METAL LOSS AND THINNING IS DUE TO OXIDATION OF CHROMIUM.

4. THINNING IS FROM INSIDE. OXIDATION ON OUTSIDE IS ALONG GRAIN BOUNDARIES.
SUMMARY

FAILURE: THE TUBE WALLS IN SEVERAL AREAS HAD GREATLY THINNED REQUIRING REPLACEMENT OF THE COIL.

SERVICE LIFE: 919 HRS SINCE REBUILDING

ENVIRONMENT: SEE ATTACHMENT FOR HRS AND TYPES OF ATMOSPHERE

PRIOR REPAIRS: COIL WAS REBUILT REPLACING ALL THIN SECTIONS, 12/74, WITH ALONIZED RA 330 AND A 9"-O" SECTION WAS REPLACED 9/75.

MATERIALS: INCOLOY 800 AND RA 330

CONCLUSIONS: THINNING WAS PROBABLY DUE TO A COMBINATION OF CARBURIZATION AND SULFIDATION. NEW COIL FABRICATED OUT OF INCONEL 702.

ACTION: REPLACED PART. ANALYSIS BY COMPANY LABS.

* * * * *

SUMMARY

FAILURE: THREE HOLES IN THREE DIFFERENT TUBE PASSES NEAR THE OUTLET END.

SERVICE LIFE: 61.5 HRS

ENVIRONMENT: SEE ATTACHMENT

MATERIAL: INCONEL 702

CONCLUSIONS: FAILURE PROBABLY DUE TO VERY SEVERE CORROSION FROM SULFUR AND/OR CARBONIZATION.

ACTION: PART REPLACED. ANALYSIS BY CONOCO LABS.

MATERIAL OF CONSTRUCTION: INCOLOY 800 causes: THE FAILURE OF THE DIPEG IS BELIEVED TO BE ASSOCIATED WITH HIGH TEMPERATURE (OFF-PROCESS) OPERATION WHICH LED TO SULFIDATION AND CARBURIZATION.

RECOMMENDATIONS
1. PROCESS TEMPERATURES MUST BE KEPT WITHIN THE DESIGN LIMITS FOR INCOLOY 800.
2. USE OF A HIGHER CHROMIUM CONTENT STAINLESS STEEL SHOULD ALLEVIATE THE SULFIDATION PROBLEM.
3. WE CONCUR WITH THE SUGGESTION BY HYGAS PERSONNEL TO ALONIZE THE FLAPPER VALVE, WHICH MAY PROVIDE IMPROVED PERFORMANCE OF THIS COMPONENT.
INTERIM REPORT, 3/72-4/74, FROM USS ENGINEERS AND CONSULTANTS, INC., TO OCR.
CLEAN COKE PROJECT

SUMMARY

3-IN CARBONIZER: SECTIONED FOR OBSERVATION AFTER 60 HRS SERVICE AT TEMP. OVER 1000°F INCLUIDING 24 HRS OVER 1300°F. EXAM. SHOWED SIGNIFICANT CARBURIZATION (CA8I0ES OF CR & IRON) AT THE SURFACE OF THE 316 S.S. SHELL. IN AREAS WHERE THE HOT SURFACE WAS IN CONTACT WITH H2S FREE FLUIDIZING GAS. THOUGH IT APPEARED TO BE STRONG AT HIGH TEMP., THE CARBURIZED STEEL WAS EXTREMELY BRITTLE WHEN COOL. DEPTH OF CARBURIZATION (20-25 MILS) WAS GREATEST FROM THE BOTTOM GAS INLET PIPE TO ABOUT THE TOP OF THE FLUID BED SECTION. ABOVE THIS POINT CARBURIZATION DECREASED RAPIDLY TO ZERO NEAR THE TOP OF THE DEENTRAINMENT SECTION. CARBURIZATION IS ATTRIBUTED TO THE ABSENCE OF H2S IN THOSE AREAS WHERE IT OCCURRED. A CONTROLLED CONCENTRATION OF H2S WILL BE MAINTAINED IN THE RECYCLE FLUIDIZING GAS TO MINIMIZE CARBURIZATION. SOME THIN WALL 316 S.S. PIPING HAS BEEN REPLACED WITH HEAVIER WALL INCOLOY 800.

CORROSION TESTS UNDER CARBONIZATION CONDITIONS: 4 SERIES OF TESTS WERE RUN USING 304 S.S., 316 S.S., INCONEL 600, INCOLOY 800, 310 S.S., AND ALONIZED 316 S.S.

SERIES 1. 1370 F, PRESS. 125 PSIA, H2S CONTENT 25-40PPM.
TEST LENGTH 13.5 HRS. 18 GRAMS OF BLACK DEPOSIT RECOVERED.

SERIES 2. 1250F, 100 PSIA, 50-100 PPM H2S. TEST LENGTH, 92 HRS. NO CARBON.

SERIES 3. SAME AS 2 EXCEPT 575 GRAMS OF CHAR PUT INTO REACTOR AROUND COUPONS. TEST LENGTH, 95 HRS. RECOVERED CHAR WEIGHED 512 GRAMS WITH NO EVIDENCE OF CARBON DEPOSITION.

SERIES 4. 1250F, 100 PSIA, NO ADDED H2S AND NO CHAR BED. TEST LENGTH, 95 HRS. H2S CONCENTRATION WAS 10-20 PPM FROM REACTIONS IN VESSEL.
6 GRAMS OF CARBON DEPOSITS RECOVERED.

MOST OF CARBON IN DEPOSITS IS IN ELEMENTAL FORM. WHEN THIS IS DEPOSITED ON AN ALLOY SURFACE IT CAN LEAD TO "METAL DUSTING." 304 S.S. SHOWED SOME EVIDENCE OF THIS. CARBURIZATION IS A FUNCTION OF BOTH H2S CONCENTRATION AND TEMPERATURE 50-100 PPM OF H2S SHOULD BE ADEQUATE TO PROTECT INCOLOY 800 FROM CARBURIZATION.

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LETTER REPORT, 2/16/70, FROM J. LETERLE, CONOCO.
HIGH TEMPERATURE CORROSION OF PARTS FROM CO2 PILOT PLANT

SUMMARY


CONCLUSIONS
1. IT APPEARS THAT SULFUR HAS REACTED WITH ALL THREE METALS TO FORM A LOW MELTING POINT EUTECTIC OF SULFUR, CHROMIUM, NICKEL, AND IRON.
2. THE 310 S.S. HAS BEEN HIGHLY CARBURIZED.
3. IF THE ENVIRONMENT THAT CAUSED THIS CORROSION IS TYPICAL THEN CAREFUL CONSIDERATION MUST BE GIVEN TO ALLOY SELECTIONS.

INTEROFFICE REPORT FROM H. CROWDER TO F. J. RADD, CONOCO, 8/25/70.
METALLURGICAL STUDY OF REACTOR AND GASIFIER VESSELS FROM THE LIGNITE GASIFICATION PRE-PILOT PLANT, CONSOL

SUMMARY

THE BENCH SCALE LIGNITE GASIFICATION PLANT, OPERATED AT LIBRARY, PA HAS EXPERIENCED HIGH TEMPERATURE CORROSION AND EMBRITTLEMENT PROBLEMS. THIS REPORT SUMMARIZES WORK ON THE FOLLOWING PARTS:
1. GASIFIER VESSEL
2. REGENERATOR VESSEL
3. REGENERATOR PRE-HEAT COIL
4. REGENERATOR THERMOWELL

CONCLUSIONS: ALL FOUR OF THE PIECES HAVE EXPERIENCED ONE OR MORE OF THE FOLLOWING PROBLEMS: HIGH TEMPERATURE SULFUR CORROSION, EMBRITTLEMENT FROM CARBURIZING, EMBRITTLEMENT FROM NITRIDING.
THESE PROBLEMS COVER THE TEMPERATURE RANGE OF 950-1900° F.
MATERIAL EVALUATION
THE PURPOSE OF THIS PROGRAM WAS TO DETERMINE WHICH MATERIALS ARE COMPATIBLE WITH MELTS TO BE FOUND IN THE MOLTEN CARBONATE PROCESS FOR REMOVAL OF SULFUR OXIDES FROM FLUE GASES.

347 S.S. IS RECOMMENDED FOR THE SCRUBBER AND THE REGENERATOR STEPS OF THE PROCESS. GNC ALLOY HAS BEEN FOUND THAT IS SUITABLE FOR THE REDUCTION STEP. SCC OF UNSTABILIZED S.S. HAS BEEN OBSERVED UNDER CONDITIONS MORE EXTREME THAN WILL BE FOUND IN THE PLANT. THEREFORE, IT IS IMPERATIVE THAT THE SCC PROGRAM BE EXPANDED TO DETERMINE WHAT THE CRITICAL PARAMETERS ARE FOR THIS MODE OF ATTACK.

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THE PURPOSE OF THIS PROGRAM WAS TO DETERMINE WHICH MATERIALS ARE COMPATIBLE WITH MELTS TO BE FOUND IN THE MOLTEN CARBONATE PROCESS FOR REMOVAL OF SULFUR OXIDES FROM WASTE GASES.

THE MOLTEN ALKALI CARBONATE EUTECTIC CONSISTS OF ESSENTIALLY EQUAL PARTS BY WEIGHT OF LITHIUM, SODIUM, AND POTASSIUM CARBONATE AND MAY BE USED TO CONTROL SO2 AIR POLLUTION BY SCRUBBING STACK GASES WITH THE MOLTEN SALT. THE RESULTANT FUSED SALT CONTAINS SULFITE WHICH DISPROPORTIONATES TO SULFATE AND SULFIDE. SUITABLE CONTAINMENT OF THESE MOLTEN SALTS IN H2O, CO2, AND H2S GASEOUS ENVIRONMENTS IS REQUIRED FOR COMMERCIAL DEVELOPMENT OF THIS TECHNIQUE TO CONTROL AIR POLLUTION. THE RESULTS OF SCREENING, MEDIUM-TERM AND LONG-TERM STATIC, DYNAMIC AND THERMAL CYCLE TESTS ARE GIVEN. AT 500°C 347 S.S. IS GOOD. AT 600°C LOW IRON, NICKEL-OR COBALT-BASED ALLOYS WITH ABOUT 23% CR PERFORM SATISFACTORILY. AT HIGHER TEMA. HIGH CR ALLOYS, CERAMICS, CERAMETS DO A GOOD JOB. MATERIALS FOR THE SMALL PILOT PLANT ARE RECOMMENDED AND THE FUTURE CORROSION PROGRAM IS OUTLINED. SEE I.N. 71 FOR ADDITIONAL DETAILS.
I. N. PROCESS SEARCH CRITERIA
73 CARBONATE MATERIAL EVALUATION

TEST REPORT BY L. GRANTHAM, ATOMICS INTERNATIONAL, 5/29/74.
SUMMARY OF FIRST YEARS OPERATION OF CORROSION AND COMPONENT TEST LOOP

SUMMARY

GOOD DESCRIPTION OF COMPONENTS USED IN THE TEST LOOP AND THEIR REACTIONS TO CORROSION, WEAR, ETC
GIVES NAMES AND EVALUATIONS.
DURING THE FIRST YEAR, THE MOLTEN CARBONATE PROCESS CORROSION AND COMPONENT TEST LOOP OPERATED 6577 HRS OR APPROX. 78% OF THE TIME. LOOP OPERATION SHOWED THAT THE CANTILEVERED CENTRIFUGAL PUMP, GRAFOIL PACKED VALVES, MODIFIED BELLows SEALED VALVES, SPRAY NOZZLE, VIEWPORT, ROOD HEATERS, RING GASKET JOINTS, COPPER GASKET JOINTS, MODIFIED RAMAPO FLOWMETER, SATURABLE CORE TEMPERATURE CONTROLLERS AND VARIOUS INSULATIONS WERE COMPATIBLE WITH THE MCP APPLICATION. HOWEVER, THE PRESSURE SENSORS, RADDI HEATERS, ORIFICES, AND WAT-PAK TEMPERATURE CONTROLLERS WERE NOT SATISFACTORY AND REQUIRE FURTHER DEVELOPMENT.
347 S.S. WAS MORE CORROSION RESISTANT TO EUTECTIC ALKALI CARBONATE-SULFIDE SULFATE MELTS AT APPROX. 500°(930°F) THAN EITHER 304 S.S. OR 321 S.S. THREE STRESS CrACKS AND SOME LOCALIZED PITTING WERE OBSERVED ON 347 S.S. COMPONENTS. WITH CONTROL OF THIS CRACKING AND Pitting, 347 S.S. WOULD HAVE A CORROSION RATE OF APPROX. 20 MILS/YEAR UNDER THESE CONDITIONS.

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I. N. PROCESS SEARCH CRITERIA
158 CARBONATE MATERIAL EVALUATION

LETTER REPORT FROM E. HOXIE TO R. SARRAF, ATOMICS INTERNATIONAL, 6/11/75.
CORROSION TEST SPOOL-6376

SUMMARY

CORROSION TEST SPOOL-6376.
TWELVE ALLOYS WERE TESTED FOR CORROSION RESISTANCE UNDER THE FOLLOWING CONDITIONS:
PROCESS UNIT: 3 GALLON RETORT AT 122 PSIG
CORROSIVE MEDIA: COAL GASIFICATION PROCESS QUENCH TANK LIQUOR
AVERAGE TEMPERATURE: 350°F
AERATION: NONE
AGITATION: NONE
LENGTH OF TEST: 9.5 DAYS
INCONEL ALLOY 600 WAS THE MOST RESISTANT ALLOY TESTED BOTH IN REGARDS TO GENERAL CORROSION RESISTANCE AND RESISTANCE TO STRESS CORROSION CRACKING.
REMARKS: NO AGITATION IN TEST VESSEL. CONSIDERABLE AGITATION WILL BE EXPERIENCED IN SERVICE. SEE I. N. 159 FOR ADDITIONAL TESTS.
CORROSION TEST SPOOL-6375

SUMMARY

SECOND GROUP OF 12 ALLOYS WAS TESTED FOR CORROSION RESISTANCE UNDER THE FOLLOWING CONDITIONS.
PROCESS UNIT: 3 GALLON RETORT AT 123 PSIG.
CORROSIVE MEDIA: COAL GASIFICATION PROCESS QUENCH TANK LIQUOR.
AVERAGE TEMPERATURE: 350F
AERATION: NONE
AGITATION: NONE
LENGTH OF TEST: 29.5 DAYS

THE DATA CONFIRM RESULTS FROM PREVIOUS TEST. SEE I.N. 158 FOR DETAILS.
INCONEL 600 RECOMMENDED FOR USE AS A CONSTRUCTION MATERIAL.

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TEST REPORT BY J.G.BRUCE, ATOMICS INTERNATIONAL, 7/23/75.
CORROSION TESTS OF MATERIALS IN HIGH TEMPERATURE MOLTEN SALT

SUMMARY

22 MATERIALS WERE CORROSION TESTED IN STATIC 1900F MOLTEN SODIUM CARBONATE (FROM THE OCR COAL GASIFICATION PROGRAM) FOR 100 HRS. THE CERAMICS AL2O3, ZR02, MGO, AND BEO AND THE METALS PLATINUM AND UNILLOY 504+1 WERE FOUND TO BE THE MOST CORROSION RESISTANT MATERIALS.
REPORT, 10/1/75, BY R. SARRAF, ATOMICS INTERNATIONAL. CORROSION TESTING OF CONSTRUCTION MATERIALS IN AI COAL GASIFICATION PROCESS QUENCH TANK LIQUOR.

SUMMARY

CORROSION TESTING OF CONSTRUCTION MATERIAL IN AI COAL GASIFICATION PROCESS QUENCH TANK LIQUOR.

CORROSION TESTS OF UP TO 90 DAYS EXPOSURE. MATERIALS TESTED INCLUDED METALS AND PLASTICS AT 350 F AND 175 F IN STRESSED AND UNSTRESSED CONDITION. QUENCH TANK CONDITIONS ARE SHOWN TO PRODUCE SCC TO SUSCEPTIBLE MATERIALS, AND A GROUP OF SUITABLE MATERIALS ARE INDICATED. FIBREGLASS REINFORCED PLASTICS ARE ALSO SHOWN TO HAVE LIMITED USEFULNESS.

1. FOR HANDLING QUENCH TANK LIQUOR AT 350 F, PRESS. TO 120 PSIG THE FOLLOWING ALLOYS HAD LOW CORROSION RATES: INCONEL 600, 601, 617, RA330, RA333.

2. FOR THE QUENCH/REGENERATOR TANK USE ABOVE MATERIALS.

3. PRELIMINARY RESULTS ON PLASTICS TESTED INDICATE SUITABILITY OF SEVERAL MATERIALS AT GREEN LIQUOR TEMPERATURES TO 200-250 F.
I.N. PROCESS Search Criteria
182 Clean Coke Material Evaluation

Interim Report, 3/72-4/74, from USS Engineers and Consultants, Inc. to OCR.
Clean Coke Project

Summary

3-in Carbonizer: Sectioned for Observation after 60 hrs service at temp. over 1000°F including 24 hrs over 1300°F. Exam. showed significant carburation (carbides of Cr & iron) at the surface of the 316 S.S. shell, in areas where the hot surface was in contact with H2S free fluidizing gas. Though it appeared to be strong at high temp., the carbured steel was extremely brittle when cool. Depth of carburation (20-25 mils) was greatest from the bottom gas inlet pipe to about the top of the fluid bed section. Above this point carburation decreased rapidly to zero near the top of the deentrainment section. Carburation is attributed to the absence of H2S in those areas where it occurred.

A controlled concentration of H2S will be maintained in the recycle fluidizing gas to minimize carburation. Some thin wall 316 S.S. piping has been replaced with heavier wall INCOLOY 800.

Corrosion tests under carburation conditions: 4 series of tests were run using 304 S.S., 316 S.S., INCONEL 600, INCOLOY 800, 310 S.S., and Alonized 316 S.S.


Series 2: 1250°F, 100 psia, 50-100 ppm H2S. Test length, 92 hrs. No carbon.

Series 3: Same as 2 except, 575 grams of char put into reactor around coupons. Test length, 95 hrs. Recovered char weighed 512 grams with no evidence of carbon deposition.

Series 4: 1250°F, 100 psia, no added H2S and no char bed. Test length, 95 hrs. H2S concentration was 10-20 ppm from reactions in vessel. 6 grams of carbon deposits recovered.

Most of carbon in deposits is in elemental form. When this is deposited on an alloy surface it can lead to "metal dusting". 304 S.S. showed some evidence of this. Carburation is a function of both H2S concentration and temperature 50-100 ppm of H2S should be adequate to protect INCOLOY 800 from carburation.

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I.N.  
183  
PROCESS  
SEARCH CRITERIA  

CLEAN COKE  
MATERIAL EVALUATION  

QUARTERLY REPORT, 1, 2, 3/75, FROM USS ENGINEERS AND CONSULTANTS, INC.  
CLEAN COKE PROJECT  

SUMMARY  
CARBONIZATION PDU: MAIN RECYCLE-GAS HEATER. 7 OF 45 ELEMENTS HAD MALFUNCTIONED (6 BURNED OUT, 1 BROKEN TERMINAL). FORMATION OF EXTENSIVE CARBON DEPOSITS ON THE HEATER ELEMENTS CAUSED BURNOUT BECAUSE OF EXCESSIVE TEMPERATURE WITHIN THE CARBON-INSULATED SHEATHS. NEW HEATER SUBASSEMBLY ORDERED WITH IMPROVED MGO INSULATION AND WIRE ARRANGEMENT TO REDUCE WATT DENSITY AND SUSCEPTIBILITY TO HEAT DETERIORATION. DURING A LATER RUN THE GAS DISTRIBUTION PLATE BECAME PLUGGED AND THE WASH OIL RECIRCULATING PUMP FAILED. CAUSES NOT DISCUSSED. PUMP REPLACED WITH A SEALED CRANE CENTRIFUGAL PUMP.  
CORROSION STUDIES: COUPONS OF VARIOUS ALLOYS WERE EXPOSED IN SEVERAL LOCATIONS IN THE PDU UNDER ACTUAL OPERATING CONDITIONS (800F AND 1300F). AT LOW TEMPERATURE ONLY CARBON STEEL SHOWED SIGNIFICANT CORROSION. AT HIGH TEMPERATURE ALONIZED INCOLOY 800 HAD A RELATIVELY HIGH CORROSION RATE. PLAIN INCOLOY 800 SHOWED NO SIGNIFICANT CHANGE AFTER 244 HRS OF HIGH TEMPERATURE TESTING. TABLES GIVE DETAILS AND RESULTS OF TESTS.  

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I.N.  
273  
PROCESS  
SEARCH CRITERIA  

CLEAN COKE  
MATERIAL EVALUATION  

MONTHLY PROGRESS REPORT, 10/75, BY USS ENGINEERS AND CONSULTANTS, INC.  
CLEAN COKE PROJECT  

SUMMARY  
CARBONIZATION PDU: RECYCLE GAS COMPRESSOR PERSISTENT PROBLEM WITH GAS LEAKS AROUND THE SHAFT BELIEVED TO BE CAUSED BY LIGHT FACE LOADING ON THE SEALS. THE REDESIGN INVOLVED CHANGING THE SEAL BALANCE FROM 70% TO 10% AND REDUCING THE SPRING FORCE BY ABOUT 20%. THIS WOULD PROVIDE GREATER FACE PRESSURE TO INSURE A MORE POSITIVE SEAL. DESIGN HAS PERFORMED SATISFACTORILY SINCE INSTALLATION (8/75).  
CORROSION TESTS IN CARBONIZATION PDU: A COMPLETE SERIES OF TEST SPECIMENS WERE EXPOSED IN THE PDU DURING OPERATION AT FLUID BED TEMP. OF BOTH 800F AND 1300F. IN LOW TEMP. EXPOSURE ONLY CARBON STEEL SHOWED SIGNIFICANT CORROSION. IN HIGH TEMP. LOCATIONS NONE OF THE CORROSION RATES WERE GREATER THAN 3.0 MPY. INCOLOY 800 SPECIMENS FROM HIGH TEMP. LOCATIONS WERE EXAMINED METALLOGRAPHICALLY AND SHOWED NO SIGNIFICANT CHANGE IN MICROSTRUCTURE. TABLES PRESENT PERTINENT DATA FOR THE TESTS.  
HYDROGENATION PDU: HIGH PRESSURE FITTINGS DEVELOPED LEAKS WHEN OPERATING TEMP. REACHED ABOUT 700F. POSSIBLY RESULT OF DIFFERENTIAL EXPANSION OF DISSIMILAR METALS (316 S.S., 304, 410 S.S. GLAND NUT, 416 S.S. COLLAR). DESIGN CHANGED TO MAKE ALL PARTS FROM 316 S.S.
SUMMARY

CARBONIZATION PDU: All corrosion test coupons from the low temperature or high temperature exposure sites were removed and evaluated. Low temperature coupons (up to 500°F) were exposed for 1566 hrs. In the gas-cooler pot only carbon steel showed significant corrosion (8.2-22.9 MPY). In the Interchanger pot carbon steel (37.5 MPY), 409 S.S. (3.9 MPY), and 430 S.S. (1.2 MPY) had the highest corrosion rates. All coupons removed from Interchanger pot bottom location generally had higher corrosion rates than previous tests.

High temperature coupons (500-1700°F) from char receivers were exposed for 850 hrs. Those in carbonizer and heater-outlet locations were exposed for 1305 hrs. Coupons generally showed small weight gains. Greatest weight losses were carbon steel and 410 S.S. in the char receivers and 304 S.S. and 314 S.S. in the heater-outlet location. All coupons from the carbonizer and heater-outlet locations showed sulfidation attack. Inconel 601 and Incoloy 800 were worst.

Tables present weight loss rates for 27 different alloys. New coupons have been installed in the carbonizer and heater-outlet locations. Sufficient data are now available for the low temperature locations and char receivers.
### I.N. PROCESS SEARCH CRITERIA

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<td>180</td>
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REPORT, NO DATE, NO AUTHOR. MATERIALS TEST PROGRAM-COED MATERIALS PROGRAM.

**SUMMARY**

**MATERIALS TEST PROGRAM**

**PYROLYSIS VESSELS:** Test coupons were welded to the inside vessel walls of the stage two pyrolysis vessel (800°F) and the stage three pyrolysis vessel (1000°F). Total test time was 4000 hours. Materials, compositions, and heat treatments of coupons given in Table XLVII. Stage two specimens—good condition visually. Stage three specimens—carbon steel coupons had experienced severe attack. Test specimens sent to Lukens Steel for metallography.

**OIL ABSORBER TOWER:** Test coupons were installed in the bottom section. Temperature was 6600-7000°F. Total test time was 6 months. INCO analysis results given in Table. Types 300 and 400 S.S. gave satisfactory results.

**HYDROTREATING PLANT:** Test coupons were installed on oil distribution plate. Temperature of 7000-8000°F and pressure of 2000-2500 PSIG. Hydrogen concentration of 90-95%. H2S concentration of 2%. Total test time of 2254 hours. INCO analysis results given in Table LII.

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### I.N. PROCESS SEARCH CRITERIA

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LETTER REPORT, 2/17/70, FROM G. CURRAN, CONSOL, TO C. SCHULZ, CONSOL. HIGH TEMPERATURE ALLOYS FOR CO2

**SUMMARY**

**MATERIAL EVALUATION OF INCOLOY 800 AND 310 S.S. IN CALCINER AT LIBRARY, PA.**

**ATMOSPHERE:** Sulfur bearing gas at high temperatures.

**APPLICATIONS**

**GASIFIER-DEVOLATILIZER INTERNALS:** Cyclones, dilegs, thermowells, pressure tap probes, heaters. Temperature range 1500-1600°F. Sulfur as H2S, 0.1%.

310 S.S. and Incoloy 800 can both be used.

**REGENERATOR INTERNALS:** Temp/Press probes, thermowells. Temperature range 1900-1950°F. Sulfur about 0.2% as SO2, H2S, S2. COS. No alloy suitable.

**EXTERNAL PROCESS PIPING:** High temperature heaters, solids transfer lines. Temperature range 1500-1950°F. Sulfur from 0-0.1% as H2S. Use 310 S.S. or Incoloy 800 equally.
I.N. PROCESS SEARCH CRITERIA
22 CO2 MATERIAL EVALUATION

LETTER REPORT, 3/4/70, FROM J. LETERLE, CONOCO, TO M. SHARRAH.
MATERIALS REVIEW CO2 PILOT PLANT

SUMMARY

REVIEW OF OPERATION AND MATERIALS EXPERIENCE IN BENCH SCALE UNIT.
REGENERATOR INTERNALS: NO SUITABLE MATERIAL AVAILABLE. USE EXPENDABLE METAL T.C. PROBE OR PERMANENT CERAMIC. WILL EXAMINE BENCH SCALE COMPONENTS AND EXPOSE COUPONS IN CO2 PLANT AND IN LAB.
USE REFRACTORY LINER, NO SUITABLE METAL AVAILABLE. MUST BE REDUCING (HIGH CO), TO ELIMINATE SLAG FUSION. 310 S.S. BETTER THAN INCOLOY 800 BUT EMBRITTLES ON CARBURIZING. SUGGEST ALONIZING FOR T.C.
DOLomite RETURN LINES: 1900F, "INERT PURGE GAS. NORMALLY DO NOT SEE SULFUR BUT SOME LEAKS IN USE 310 S.S. INSTEAD OF INCOLOY 800 TO REDUCE SULFIDATION. PURGE GAS CONTAINS 3-4% CO THAT WILL CARBURIZE AND EMBRITTLE 310 S.S. MUST MODIFY GAS.
COOLING WATER SYSTEMS: USE INHIBITOR (DICHROMATE) WITH CARBON STEEL.
TO PREVENT SCC-CL DO NOT ALLOW WATER TO STAND IN PIPES ON SHUTDOWN.
WELDING: AVOID HIGH NI WELD RODS.
MISC.: BENCH SCALE TESTS SHOW CORROSION OF 310 S.S. AND HASTELLOY X AT 1400 TO 1500F. NO REASON.

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I.N. PROCESS SEARCH CRITERIA
43 CO2 MATERIAL EVALUATION

INTEROFFICE REPORT, 4/10/70, FROM J. LETERLE TO J. KIRK, CONOCO.
MATERIALS RESEARCH PROGRAM FOR CO2

SUMMARY

MATERIALS RESEARCH PROGRAM FOR CO2
SET UP THREE PHASE EVALUATIONS:
1) FAILURE ANALYSIS OF BENCH SCALE COMPONENTS.
2) HIGH TEMP. CORROSION, SULFIDATION, CARBURIZATION STUDY OF MATERIALS.
3) EXPOSURE OF COUPONS IN PILOT PLANT.
REPORT GIVES DETAILS OF PROGRAM.
**SUMMARY REPORT FROM F. RADO TO J. KIRK, PONCA CITY, 9/4/70.**

**PRESENT STATUS SUMMARY OF CONSOL, RAPID CITY METALLURGY RESEARCH BACKUP STUDIES**

**MAJOR METALLURGICAL PROBLEMS OF CO₂ PLANT**

1. **HIGH TEMPERATURE SULFIDE ATTACKS**
2. **CARBURIZATION AND OR OXIDATION.**
3. **CO₂ CAN PRODUCE CARBURIZATION OF CR BEARING STEELS.**
4. **INCOLOY 800 TUBES IN GAS HEATERS (B-207) SHOULD BE CLOSELY INSPECTED AT SHUTDOWNS.**
5. **ONE OF THE MAJOR UNRESOLVED PROBLEMS IS THE ULTIMATE LONG-LIVED MATERIALS FOR THE THERMOWELLS AND THE SAMPLING TUBES.**
6. **A CONTINUOUS, ON-STREAM INSPECTION IS VITAL TO A SUCCESSFUL LINING OUT AND SMOOTH NORMAL OPERATION OF THIS PLANT.**

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**LAB DATA FROM L. RICHMOND, STEARNS-ROGER, TO C. SCHULZ, CONOCO.**

**OPERATING CONDITIONS—RAW LAB DATA**

**SUMMARY**

**OPERATING CONDITIONS ON CORROSION. RAW LAB DATA OF OPERATING CONDITIONS COVERING RECENT RUNS TO BE USED FOR DETERMINING THE EFFECT OF OPERATING CONDITIONS ON CORROSION. ALL AVAILABLE LAB ANALYSES AND OPERATING TEMPERATURES ARE ATTACHED.**
BENDING PROPERTIES AND HIGH TEMPERATURE SULFUR CORROSION RESISTANCE OF ALONIZED INCOLOY 800.

SUMMARY

BENDING PROPERTIES AND HIGH TEMPERATURE SULFUR CORROSION RESISTANCE OF ALONIZED INCOLOY 800.

CONCLUSIONS

1. The alonized layer on Incoloy 800 is made up of two parts, a hard brittle outside layer and a more ductile inner layer. The inner layer provides the most reliable protection against corrosion.

2. An annealing treatment of 1830F for 24 HRS makes the outer alonized coating more ductile.

3. In the annealed condition tubing could be bent to a radius of 3.75 in at 1650F, a radius of 6 in at 1300F, or a radius of 26 in at 1000F.

4. A sound alonized layer gives protection against high temperature H2S corrosion.

5. Bending, in either the annealed or unannealed condition, opens up cracks in the outer layer, thereby reducing to some degree the corrosion resistance of the surface.

6. Bending in the "as-alonized" condition or bending in the annealed condition at too low a temperature will also crack the inner layer and greatly reduce the corrosion resistance of the alonized coating.

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I.N.  PROCESS    SEARCH CRITERIA
135  CO2        MATERIAL EVALUATION

REPORT, 6/30/75, TO CONSOLIDATED COAL CO. FROM S. DAKOTA SCHOOL OF MINES & TECH. CORROSION OF MATERIALS IN CONTACT WITH CACO3-Ca(OH)2 MELTS.

SUMMARY
A STUDY WAS INITIATED TO IDENTIFY THOSE MATERIALS WHICH WOULD FUNCTION WELL AS MELT CONTAINERS. SPECIFIC AREAS INVESTIGATED WERE: 1) GROWTH OF THE OXIDE LAYER ON ALLOY STEELS. 2) WEIGHT LOSS OF METAL SAMPLES IN CONTACT WITH MELTS. 3) MICROSTRUCTURE OF OXIDIZED ALLOYS AND OXIDATION PRODUCTS.

CONCLUSIONS
1. WEIGHT LOSS MEASUREMENTS INDICATE THAT THE S.S. CORRODE PRIMARILY THROUGH SPALLING OF THE PROTECTIVE OXIDE LAYER.
2. BECAUSE CORROSION RATE DECREASES AS THE CR CONTENT OF THE S.S. INCREASES HIGH CR ALLOYS, IE, TYPES 309, 310, AND 446 ARE BEST SUITED AS MELT CONTAINERS.
3. THE ALLOYS WHICH CONTAIN RELATIVELY LARGE AMOUNTS OF NICKEL, IF, INCONEL 600 AND 601 AND NICKEL 20C WOULD NOT FUNCTION WELL AS MELT CONTAINERS EITHER BECAUSE OF DEVELOPMENT OF SURFACE POROSITY OR BECAUSE OF HIGH CORROSION RATES.

I.N.  PROCESS    SEARCH CRITERIA
56  CO2        MATERIAL EVALUATION

REPORT, 10/20/75, BY H. CREWDER, CONOCO. CORROSION COUPON EVALUATION, SERIES III.

SUMMARY
EVALUATION OF CORROSION OF SEVERAL ALLOYS IN REGENERATOR GAS.
EXPOSURE: SERIES II - 550 HRS, OXIDIZING
650 HRS, REDUCING
SERIES III - 500 HRS, OXIDIZING
620 HRS, OXIDIZING

VERY DETAILED METALLOGRAPHIC ANALYSIS. CORROSION RATES 10-360 MPY.
INCONEL - 706, 718, 793. INCOLOY - 800, 801.
LETTER, 11/7/75, FROM J. LETERLE, CONOCO, TO C. SCHULZ, LIBRARY.

THIRD SERIES OF METAL COUPON EXPOSURE TESTS

SUMMARY

SERIES II-1200 HRS EXPOSURE, RUN 19, 20, 21
SERIES III-ADDITIONAL 1100 HRS, RUNS 22, 23, 24
SUMMARY: 1) HAYNES 150 ALLOY EXCELLENT COMPARED WITH HAYNES 133 AND 25 DIFFERENT COBALT BASED ALLOYS. 2) ADDITIONAL ANALYTICAL WORK TO BE DONE.

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REPORT, N.D., BY J. DEVAN, R. COOPER, W. HARRIS, ORNL.
MATERIAL EVALUATION—HEAT EXCHANGER TUBES

SUMMARY

MATERIAL SELECTION FOR HEAT EXCHANGER TUBES IN A MODULAR INTEGRATED UTILITY SYSTEM

TEST PROGRAM: SURVEILLANCE COUPONS OF 310 AND 316 S.S., INCOLOY 800 AND 825 WERE EXPOSED IN COMBUSTION POWER COMPANY'S MODEL FLUIDIZED BED REACTOR.
TEST CONDITIONS: TEMP. 1600-1700 F, SEVERAL HUNDRED PERCENT EXCESS AIR, NO COOLING TUBES IN BED. DOLOMITE ADDED TO TIE UP S02 AND A GEORGIA CLAY ADDED AS A CORROSION INHIBITOR. SPECIMENS LOCATED IN THE FREE BOARD POSITION JUST ABOVE THE FLUID BED. INCOLOY 825 WAS EXPOSED FOR 1000 HRS. 310 AND 316 S.S. AND INCOLOY 800 WERE EXPOSED FOR 500 HRS AND 1000 HRS.
RESULTS: SIGNIFICANT INTERGRANULAR OXIDATION OCCURRED ON ALL FOUR MATERIALS. SCALE WAS THICKEST ON 316 S.S. NO INDICATION OF SULFIDATION OR CARBURIZATION. ANALYSIS OF DATA WILL CONTINUE.
FUTURE: CONTROLLED EXPERIMENTS DUPLICATING THE CONDITIONS EXPECTED IN THE MIUS SYSTEM ARE NEEDED TO ASSES COMPATIBILITY PROBLEMS.
I.N. PROCESS SEARCH CRITERIA
1 HYGAS MATERIAL EVALUATION

LETTER, 4/2/75, FROM A. MAC NAB, C. F. BRAUN, TO P. TARMAN, IGT
MATERIAL SELECTION FOR HYGAS STEAM/IRON GASIFICATION PILOT PLANT

SUMMARY

DISCUSSION OF MATERIALS TO RESIST SULFIDATION IN PIPING TO THE REACTOR AND SECOND STAGE REDUCER SPARGE RING. ATMOSPHERE WILL BE REDUCING GAS CONTAINING 0.1 MOLE PERCENT H2S AT 2000°F. INCOLOY 800 AND 310 S.S. WILL NOT HOLD UP.
RECOMMEND 50CR-50NI (ASTM A560 OR UNILOY 50/50) OR ALONIZED 310 S.S. OR ALONIZED INCOLOY 300. 50CR-50NI HAS GOOD SULFIDATION RESISTANCE BUT POOR HIGH TEMP. STRENGTH. RECOMMEND USE OF THIS MATERIAL AS CLADDING TO A SUBSTRATE OF INCOLOY 800 OR 310 S.S.

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I.N. PROCESS SEARCH CRITERIA
79 HYGAS MATERIAL EVALUATION

LETTER, 6/2/75, FROM P. TARMAN, IGT, TO A. MAC NAB, C. F. BRAUN
MEMO, 5/30/75, FROM R. BACHTA TO P. TARMAN, IGT
FEASIBILITY AND AVAILABILITY OF INCOCLAD 671/800 PIPE

SUMMARY

TARMAN COMMENTS ON MAC NAB’S SUGGESTION OF USING 50CR-50NI AS CLADDING ON INCOLOY 800 AND HIS RECOMMENDATION THAT IGT CONTACT HUNTINGTON ALLOYS FOR INFORMATION.
NMEMO FROM BACHTA GIVES INFORMATION FROM HUNTINGTON ALLOYS. PROBLEMS IN GETTING THIS MATERIAL WOULD BE 1) SIZE, BIGGEST PRODUCED BY HUNTINGTON IS 2-1/2” OD, IGT NEEDS 4” OD. 2) CLADDING THE INTERIOR OF THE PIPE. MOST PRODUCTS CLAD ON OUTSIDE ONLY.
INCOCLAD 671/800 PIPE HAS AN INCOLOY 800 SUBSTRATE WHICH PROVIDES STRENGTH AT HIGH TEMPERATURES AND INCONEL 671 (APPROX. 50CR-50NI) CLADDING WHICH PROVIDES RESISTANCE TO SULFIDATION.
I.N. PROCESS SEARCH CRITERIA
250 HYGAS MATERIAL EVALUATION

LETTER, 7/30/75, FROM A. MAC NAB, C. F. BRAUN, TO H. FRANKEL, ERDA
INSPECTION OF HYGAS PILOT PLANT BEFORE USING EASTERN BITUMINOUS COAL

SUMMARY

HYGAS IS PREPARING TO CHANGE ITS FEEDSTOCK TO EASTERN BITUMINOUS COAL. IT'S EXPECTED THAT THESE HIGHER SULFUR COALS WILL BE MORE CORRODIVE THAN LIGNITE AND THE CHANCES FOR COMPONENT FAILURE WILL BE INCREASED. THEREFORE IT IS IMPERATIVE TO HAVE AS THOROUGH AN UNDERSTANDING OF THE PRESENT STATE OF THE EQUIPMENT AS POSSIBLE. THIS SHOULD INCLUDE SECTIONING OF CRITICAL COMPONENTS TO ESTABLISH DEPTH OF PENETRATION BY CORROSION. ACTION IS BEING TAKEN TO IMPLEMENT THIS PROGRAM.

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I.N. PROCESS SEARCH CRITERIA
255 HYGAS MATERIAL EVALUATION

TELECON, 12/22/76, FROM S. DANYLUK, ANL, TO B. ORCHARD, IGT.
STATUS OF FAILURE ANALYSES OF FAILED COMPONENTS FROM HYGAS PILOT PLANT

SUMMARY

1. PROTECTION TUBE FROM AAG.
   ANALYSIS HAS SHOWN THAT THE 310 S.S. THERMOWELL WAS MELTED AT THE END OF THE TUBE WHICH WAS INSIDE THE GASIFIER. CAUSE OF FAILURE WAS EXCESSIVE DEVIATION FROM SPECIFIED TEMPERATURES.

2. 1/4" NITROGEN PURGE LINE.
   EMBRITTLEMENT OF THE GRAIN BOUNDARIES IS VERY SEVERE AND THE TUBE FAILED BY BRITTLE FRACTURE. SULFUR CONTENT ON INNER PHASE OF TUBE IS 1-2%. ORIGIN OF SULFUR UNKNOWN.

3. ALONIZED 316 S.S. AND 446 S.S. COUPONS.
   EVALUATION OF GASIFIER COUPONS IS UNDERWAY.

33
LETTER W/PHOTOS, 1/25/77, FROM S. DANYLUK, ANL, TO S. DAPKUNAS, ERDA.

ELECTRON MICROPROBE DATA-316 S.S. "ALONIZED" COUPONS FROM GASIFIER

SUMMARY

TWO COUPONS HAVE BEEN ANALYSED. ONE FROM RUN 61 AND A LONGER EXPOSURE COUPON FROM RUN 67. THE DATA SHOW THE NICKEL IS DEPLETED AND SULFUR AND ALUMINUM ARE ENRICHED IN THE SURFACE AREA FOR RUN 61. THESE EFFECTS ARE EXAGGERATED AND GRAIN BOUNDARY PENETRATION OF SULFUR IS MORE EVIDENT AFTER RUN 67. ANALYSES OF THESE SURFACE TREATED SPECIMENS IS BEING CONTINUED AND PROGRESS WILL BE REPORTED.

*   *   *   *   *
INVESTIGATION OF PERFORMANCE OF BOILER TUBES IN A FLUID BED COMBUSTION SYSTEM. SPECIMENS WERE TESTED IN THREE LOCATIONS: IDENTIFIED AS VERTICAL WATER-COOLED BUNDLE (VWCB), LOWER TUBE BUNDLE (LTB), UPPER TUBE BUNDLE (UTB). TUBE TEMPERATURES WERE BETWEEN 1100-1500°F.

OPERATING CONDITIONS: BITUMINOUS COAL WAS BURNED IN A BED OF LIMESTONE AT A FLUIDIZING VELOCITY OF 5.7 FT/SEC. AVG. OPERATING CONDITIONS WERE: COAL RATE, 61.3 LB/HR; LIMESTONE RATE, 20 LBS/HR; EXCESS AIR, 13.6%; BED TEMP., 1545°F. AVERAGE S02 EMISSION WAS 230 PPM. AVERAGE CARBON UTILIZATION WAS 86%.

TEST WAS TERMINATED AFTER 482 HRS WHEN THE CARBON STEEL TUBE IN LOWER BUNDLE FAILED.

- CARBON STEEL: UTB-SEVERELY CORRODED. LTB MIDDLE ROW-COMpletely REACTED. VWCB-DESTROYED COMPLETELY.

410 S.S.: UTB-THIN LAYER OF CORROSION WITH COARSER CARBIDES IN STRUCTURE THAN ORIGINAL. LTB-HEAVY CORROSION WITH COARSE CARBIDES IN GRAIN BOUNDARIES. VWCB-TUBE DESTROYED.

446 S.S.: UTB-NO CORROSION. LTB-SOME SIGMA PHASE IN MICROSTRUCTURE WITH GRAIN GROWTH ON OUTER SURFACE. VWCB-TUBE FAILED VIA A CORROSION CRACK.

304 S.S.: UTB-NO CORROSION, NO PRECIPITATES. LTB-SOME GRAIN GROWTH WITH PRECIPITATION AT GRAIN BOUNDARIES. VWCB-INTERGRANULAR CORROSION WAS PRESENT.

316 S.S.: UTB-NO CORROSION, NO PRECIPITATES. LTB-MORE CORROSION THAN THE G304 S.S. TUBES IN THIS POSITION WITH PRECIPITATES AT THE GRAIN BOUNDARIES. VWCB-LEAST CORROSION OF ANY TUBES TESTED.

CARBON STEEL, 410 S.S., AND 446 S.S. FROM VWCB HAD INTERNAL DEPOSITS WHICH PROBABLY ALLOWED THE TUBES TO OVERHEAT. THESE TESTS ARE PROBABLY NOT VALID.

REPORT CONTAINS PHOTOMICROGRAPHS OF SAMPLES.

* * * * *
FLUIDIZED BED COMBUSTION-CORROSION STUDIES

SUMMARY

THE THIRD AND LAST 500 HR TEST WITH THE CORROSION/EROSION COMBUSTOR WAS COMPLETED. TEST LENGTH, 505 HRS.

THE TUBES IN LOWER HORIZONTAL BUNDLE APPEARED TO BE IN GOOD CONDITION. THE TEMP. SHOULD NOT HAVE EXCEEDED 1100 F (NOT MEASURED). TUBES IN UPPER HORIZONTAL BUNDLE HAD NO INDICATION OF EXTERNAL CORROSION. WATER COOLED VERTICAL TUBES WERE COATED WITH A BLACK DEPOSIT THAT PROBABLY WAS LARGELY CARBON.

PHOTOMICROGRAPHS INCLUDED OF TUBES FROM 1ST SERIES OF TESTS. THESE ARE IN ADDITION TO THE ONES PRESENTED IN QUARTERLY REPORT (10-12/71).

SEE I.N. 372 FOR FURTHER INFORMATION.

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I.N. PROCESS SEARCH CRITERIA
374 MERC MATERIAL EVALUATION

QUARTERLY REPORT, 1/1-3/31/72, FROM MORGANTOWN ENERGY RESEARCH CENTER.

FLUIDIZED BED COMBUSTION-CORROSION STUDIES

SUMMARY

THE THIRD AND LAST OF THE LONG DURATION TESTS WITH THE CORROSION/EROSION COMBUSTOR WAS COMPLETED WITH A TOTAL TEST TIME OF 505 HRS. THE TUBES WERE PHOTOGRAPHED AND ARE BEING PREPARED FOR SHIPMENT TO ALBANY, OREGON FOR EXAM.

SEE I.N. 374 FOR ADDITIONAL DETAILS.
MONTHLY REPORT, 4/72, FROM MORGANTOWN ENERGY RESEARCH CENTER.
FLUID-BED COMBUSTION-CORROSION STUDIES

SUMMARY

RESULTS FROM SECOND LONG-DURATION CORROSION/erosion TESTS ARE PRESENTED.
LTB—LOWER TUBE BUNDLE, UTB—UPPER TUBE BUNDLE, VWCB—VERTICAL WATER-COOLED BUNDLE.
SEE I.N. 372 FOR OPERATING CONDITIONS.

CARBON STEEL (SEAMLESS): VWCB—NO CHANGE FROM ORIGINAL. UTB—SLIGHT INTERGRANULAR CORROSION ON EXTERNAL SURFACE.

410 S.S.: VWCB—MINOR EXTERNAL SURFACE CORROSION. LTB—MIDDLE TUBE HAS SIGNS OF SURFACE DECARBURIZATION, COARSENING OF GRAIN STRUCTURE, Pitting and scaling of surface.

416 S.S.: VWCB—UNCHANGED. LTB—ALL TUBES HAD SOME GRAIN GROWTH AT SURFACE WITH SLIGHT AMOUNT OF SURFACE CORROSION. UTB—VERY SLIGHT SURFACE CORROSION.

304 S.S. (WELDED): LT—SURFACE CORROSION. SOME PRECIPITATED CARBIDES. UTB—MODERATE INCREASE IN GRAIN SIZE.

304 S.S. (SEAMLESS): VWCB—INCREASE IN GRAIN SIZE. LTB—INCREASE IN GRAIN SIZE WITH SURFACE CORROSION AND PRECIPITATED CARBIDES AT GRAIN BOUNDARIES. UTB—INCREASE IN GRAIN SIZE WITH SLIGHT INTERGRANULAR CORROSION AT SURFACE.

316 S.S. (WELDED): VWCB—NO CHANGE. UTB—INCREASE IN GRAIN SIZE AND SLIGHT CORROSION AT THE SURFACE.

316 S.S. (SEAMLESS): VWCB—NO CHANGE. LTB—SLIGHT CORROSION AT SURFACE WITH CARBIDE PRECIPITATES AT GRAIN BOUNDARIES. UTB—SLIGHT INCREASE IN GRAIN SIZE AND SOME SURFACE CORROSION.

PHOTOMICROGRAPHS OF CORRODED SAMPLES INCLUDED IN REPORT.
CALCULATIONS ARE CONTINUING TO DETERMINE TUBE METAL TEMPERATURES AT THE INLET AND OUTLET OF EACH TUBE.
SEE I.N. 372 AND 374 FOR ADDITIONAL TESTS.
PAPER, 12/1/71, BY R. BORIO & R. HENSEL, COMBUSTION ENGINEERING.
COAL-ASH COMPOSITION AS RELATED TO HIGH TEMPERATURE FIRESIDE CORROSION
AND SULFUR OXIDES EMISSION CONTROL

SUMMARY

COAL-ASH COMPOSITION AS RELATED TO HIGH TEMPERATURE FIRESIDE CORROSION AND
SULFUR OXIDES EMISSION CONTROL.
THIS PAPER EVALUATES VARIOUS COALS IN TERMS OF CORROSION POTENTIAL AND
SHOWS HOW HIGH TEMPERATURE CORROSION CAN BE REDUCED BY INTRODUCING PROPER
AMOUNTS OF ADDITIVES. THE EFFECTS OF ADDITIVES ON AIR POLLUTION AND ASH
DEPOSITION ARE DISCUSSED.

LETTER, 8/20/75, FROM J. KELLY, ROLLED ALLOYS, TO H. FRANKEL, ERDA.
HOT CORROSION IN INCOLOY 900 COMPONENTS

SUMMARY

QUESTIONS CONCLUSIONS IN CONOCO TECHNICAL SERVICE REPORT 79-74-1301
DESCRIBING A HOT CORROSION PROBLEM IN ALLOY 800 (INCOLOY).
1. DOES NOT BELIEVE THAT THERE IS A GAMMA-TO-ALPHA PHASE CHANGE IN AREAS
DEPLETED OF CHROMIUM BY INTERNAL OXIDATION. HE BELIEVES THAT THE
RESULTING NI RICH MATRIX WILL BE EVEN MORE STABLE THAN THE ORIGINAL
ALLOY WITH RESPECT TO TRANSFORMATION.
2. ONE OF THE REMEDIAL MEASURES WAS TO INCREASE THE GRAIN SIZE TO MINIMIZE
CARBURIZATION ATTACK. HIS EXPERIENCE INDICATES THAT FINER GRAINED
MATERIALS ARE MORE RESISTANT TO CARBURIZATION THAN ARE COARSE GRAINED
MATERIALS.
I.N. PROCESS SEARCH CRITERIA
26 SRC MATERIAL EVALUATION

LETTER, 3/19/74, FROM A. HENKE, GULF R&D, TO B. SCHMID, SRC.
MATERIALS MONITORING PROGRAM FOR SRC PROCESS
LETTER REPORT, 1/30/74, FROM J. MCCOY, GULF R&D, TO B. SCHMID, SRC.
CORROSION/EROSION MONITORING PROGRAM AT SRC

SUMMARY

ESTABLISHMENT OF A CORROSION/EROSION MATERIALS MONITORING PROGRAM AT SRC
PLANT. PROGRAM CONSISTS OF:
1. ULTRASONIC TESTING TO DETERMINE THICKNESS AT KEY POINTS IN THE EQUIPMENT.
2. VISUAL INSPECTION.
3. EVALUATION OF TEST COUPONS OF: 310 S.S., 321 S.S., 304 S.S., MONEL,
INCOLOY 800, CARBON STEEL, STELLITES, COLMONOY, TUNGSTEN CARBIDE,
321 S.S. (ALUMINIZED).

I.N. PROCESS SEARCH CRITERIA
267 SRC MATERIAL EVALUATION

REPORT, 5/28/76, FROM D. CANFIELD TO L. SAMUELS, FT. LEWIS.
FAILURE AND REPAIR OF THE WASH SOLVENT COLUMN

SUMMARY

FAILURE REPORT: THE SHELL OF THE 3/8" CARBON STEEL (SA-515 GR. 55) COLUMN
STARTED TO LEAK BETWEEN THE 19TH AND 19TH TRAYS. ATTEMPTS TO PATCH THE
HOLE FAILED. THE TRAYS WERE REMOVED AND THE COLUMN WAS INSPECTED WITH
THE FOLLOWING RESULTS. 1) NEARLY ALL THE TRAY SUPPORT RINGS AND DOWNSOMER
Supports FROM TRAYS 12-21 WERE ALMOST COMPLETELY CORRODED, 2) AREAS OF
LIQUID PHASE CONTACT WERE CORRODED MORE THAN THOSE IN CONTACT WITH VAPOOR
PHASE, 3) 14 GAUGE 304 S.S. TRAYS HAD LOST ABOUT 7 MILS, 4) THE 16 AND 20
GAUGE 304 S.S. VALVES WERE THINNING AT CLIP ENDS, 5) THERE APPEARED TO BE
NO CORROSION PRODUCTS PRESENT.

FAILURE ANALYSIS: THE CORROSION APPEARS TO BE SIMILAR TO NAPTHENIC ACID
CORROSION AS DESCRIBED IN METALS HANDBOOK, VOL. 1. HOWEVER, NO DEFINITE
CONCLUSIONS ARE MADE.

REPAIRS: THE TOP 18-FT SECTION OF THE 32-IN OD COLUMN WAS REPLACED USING
1/4-IN 316L S.S. THE TRAYS AND DOWNSOMER SUPPORTS WERE REPLACED WITH
316L S.S. NEW 316 S.S. TRAY VALVES WERE INSTALLED.

CORROSION TEST RESULTS: CORROSION RACKS WERE INSTALLED IN WASH SOLVENT
COLUMN TO EVALUATE MATERIALS. TYPE 321 S.S. AND INCOLOY 800 SHOWED VERY
GOOD RESISTANCE TO THE CORROSIVE ENVIRONMENT. MOLY BEARING ALLOYS (2-3%) ARE
THOUGHT TO HAVE GOOD CORROSION RESISTANCE TO NAPTHENIC ACID CORROSION.
SEE I.N. 302 FOR FURTHER INFORMATION.
TELECON, 11/29/76, FROM S. DANYLUK, ANL, TO B. MASSA, LUMMUS.
SUBSTITUTE MATERIALS FOR 310 S.S. TO BE USED IN GASIFIER.

SUMMARY

310 S.S. WAS RECOMMENDED AS A SUBSTITUTE FOR INCOLOY 800 THERMOCOUPLE SHEATHS WHICH WERE FAILING IN SERVICE. HOWEVER, 310 S.S. SHEATHS WILL NOT BE AVAILABLGE FOR SEVERAL MONTHS. 446 S.S. WAS RECOMMENDED BUT EMBRITTLEMENT MAY BE A PROBLEM.

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METAL DUSTING
CARBONIZATION PDU: LEAKS IN THE LARGE FLANGES OF THE REACTOR. BELIEVED TO BE A LOSS OF COMPRESSION ON THE RING SEALS BECAUSE OF VERY SLIGHT ELONGATION OF THE STUD BOLTS AFTER MORE THAN 850 HR. EXPOSURE TO TEMP. IN THE RANGE 1300-1400°F POSSIBLY DUE TO STRESS RELAXATION. BOTH THE CARBONIZER & CYCLONE WERE IN EXCELLENT CONDITION AFTER 1724 HRS. OF PDU OPERATION (863 HRS. AT 1300°F AND HIGHER).

THREE INCH CONTINUOUS CARBONIZER: PERSISTENT BURNOUT OF THE HEATING ELEMENTS OF THE RECYCLE GAS HEATER. THESE HEATERS WERE REPLACED WITH HIGHER CAPACITY HEATERS. CAUSE OF HEATER FAILURE WAS METAL DUSTING. H2S LEVELS OF 50-100 PPM HAVE BEEN INTRODUCED INTO THE FLUIDIZING GAS TO INHIBIT THE METAL DUSTING. ALSO 6-8% WATER IS INJECTED INTO THE GAS TO INHIBIT CARBON DEPOSITION AND TO GASIFY A PORTION OF THE DEPOSITS THAT FORM.

BINDER PREPARATION PDU: GEAR PUMP (PDP-401) WAS REPLACED BECAUSE OF EROSION BY CHAR PARTICLES IN THE WASH OIL. THIS PUMP WAS USED FOR TRANSFER OF FEED TO THE FEED TANK. REPLACED BY A DRUM TRANSFER PUMP WHICH IS BETTER SUITED FOR THE ABRASIVE SERVICE. TOPPED TAR PUMP (PDP-403). LOSING PRESSURE. LOOSE IMPELLOR DAMAGED PUMP SHAFT. CHAR FINES & PROCESS FLUID HAD ERODED THE BEARINGS. REPLACED ALL DAMAGED COMPONENTS & MODIFIED PUMP WITH A LIP SEAL TO KEEP PARTICULATES AWAY FROM THE BEARINGS.
PROCESS SEARCH CRITERIA

FAILURE REPORT, 4/16/74, FROM L. WOLFE, CONOCO, TO J. LETERLE, CONOCO.
MICROPROBE AND METALLOGRAPHIC STUDIES OF PITTED FURNACE TUBES.

SUMMARY

MICROPROBE AND METALLOGRAPHIC STUDIES OF PITTED FURNACE TUBES. THE LIGNITE GAS PLANT HAS HAD SEVERE CORROSION PROBLEMS FROM HIGH TEMP. SULFUR. A ZINC OXIDE SYSTEM WAS INSTALLED FOR REMOVING SULFUR. Two runs totaling 150 HRS were made with very low sulfur gas. During these runs the inside of the furnace tubes were severely pitted and one burst.

CONCLUSIONS

1. THE PITS ARE IN CARBURIZED METAL.
2. ORIGINAL SURFACE HAS SOME SULFUR CORROSION WHICH SEEMS TO RETARD THE CARBURIZATION AND PITTING.
3. MOST PIT SURFACES ARE COVERED WITH BLACK, MAGNETIC SCALE WHICH IS OFTEN IN TWO DISTINCT LAYERS.
4. THE OUTER, THICKER SCALE IS PARTICLES OF METAL AND METAL OXIDES.
5. THE INNER SCALE IS A THIN LAYER OF CR-RICH OXIDE WHICH CONTAINS SOME IRON.
6. TUBES OF INCOLOY 800 AND 321 S.S. PITTED SIMILARLY.
7. THIS ATTACK LOOKS SIMILAR TO "METAL DUSTING" WHICH DESCRIBES THE SCALE AS GRAPHITE WITH METAL PARTICLES. THE RAPID CITY SCALE IS CR-DEPLETED METAL PARTICLES WHICH ARE PARTIALLY OXIDIZED.

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I.N. PROCESS SEARCH CRITERIA

101 CO2 PIPING

OFFICIAL MINUTES OF CONSOL PILOT PLANT COIL CORROSION MEETING, 5/1/74.
PREPARED BY S.W. WORDEN.

SUMMARY

OFFICIAL MINUTES OF CONSOL PILOT PLANT COIL CORROSION MEETING, 5/1/74.
PRIMARY SUBJECT WAS CORROSION OF THE GASIFIER PREHEATER COILS AND HOW TO REDUCE THE RATES OF ATTACK. THE SULFIDATION PROBLEM WAS CURED BY INSTALLING A DESULFURIZER IN THE SYSTEM. THIS, HOWEVER, INTRODUCED A NEW TYPE OF CORROSION CALLED "METAL DUSTING" WHICH IS NORMALLY CONTROLLED BY A LOW LEVEL SULFUR CONTENT IN THE GAS. RESEARCH INTO THE PROBLEM WAS RECOMMENDED FROM THE MATERIALS EVALUATION VIEWPOINT AND 2) CHANGING PILOT PLANT PROCEDURE.
APPENDICES-SUMMARY OF SHUTDOWN CAUSES WITH DETAILS.
OPERATING CONDITIONS FOR 6 COILS.
I.N. PROCESS SEARCH CRITERIA
96 CO2 PIPING

INFORMAL NOTES, 5/1/74 BY H. CEIR, OCR, CONOCO CORROSION CONFERENCE.

SUMMARY

PROBLEM AREA: HEATER TUBES IN GASIFIER RECYCLE GAS SERVICE.

CONSSENSUS WAS THAT THE KEY TO SATISFACTORY INTERIM PERFORMANCE OF THE HEATER TUBES WAS THE FORMATION AND MAINTENANCE OF AN IMPERVIOUS FILM ON THE TUBE SURFACE. SUGGESTED WAYS, 1) SULFIDING, 2) OXIDIZING, 3) ALONIZING, 4) COATING OTHER THAN ALONIZING. ALL OF CONOCO'S METALLURGICAL STAFF & A. MACNAB FAVORED SULFIDATION. R. PERKINS, C. FINK, H. CEIR FAVORED OXIDATION.

PROS AND CONS OF EACH DISCUSSED. (OFFICIAL MINUTES TO BE PREPARED BY S. WORDEN, CONOCO).

SEE I.N. 101 FOR OFFICIAL MINUTES.

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I.N. PROCESS SEARCH CRITERIA
102 CO2 PIPING

FAILURE REPORT FROM F. RADD TO C. FINK, CONOCO, 5/30/74.

INCOLOY 800 HEATER COILS

SUMMARY

HIGH TEMPERATURE CORROSION CAUSATIVE AND CONTROL ANALYSIS FOR INCOLOY 800 ALLOY HEATER COILS. MANY ALLOYS ARE SUBJECT TO AN EXTREMELY VIRULENT FORM OF HIGH TEMPERATURE PITTING ATTACK ("METAL DUSTING"). AN INCOLOY 800 HEATER COIL SUFFERED A HIGH TEMP. CORROSION FAILURE WITH DEEP SPHERICAL PITS WHICH COVERED 30-40% OF THE SURFACE. THIS COIL WAS SUBJECTED TO AN ASSESSMENT OF THE MAJOR CONTROLLING VARIABLES AND THE PRINCIPAL CAUSATIVE MECHANISMS INVOLVED THAT COULD LEAD TO SUCH AN ATTACK. METALLOGRAPHIC, X-RAY DIFFRACTION, AND X-RAY MICROPROBE ANALYSES WERE USED IN THE EXAMINATION OF THE COIL. EXPLANATIONS ARE GIVEN TO EXPLAIN THE CAUSE OF THE "METAL DUSTING" PHENOMENON.
OXIDATION
GOOD PROCESS DESCRIPTION OF THE WESTINGHOUSE COAL GASIFICATION SYSTEM.

FAILURES:
1. NOZZLE PLATE AND NOZZLE EXPERIENCED A SEVERE FORM OF HIGH TEMP. CORROSION BY CARBURIZATION AND OXIDATION DUE TO EXCESSIVE TEMP. AND ALTERNATING REDUCING-OXIDIZING ENVIRONMENTS.
2. MELTING OF SHROUD MATERIAL WAS RESULT OF LOCAL OVERHEATING CAUSED BY CRACKING OF THE NOZZLE PLATE-PILOT INLET HOUSING WELD, PROBABLY MALFUNCTION OF SEVERAL NOZZLES, AND ACCUMULATION OF HEAVY CARBON DEPOSITS AHEAD OF THE BURNERS.
3. CRACKING IN THE SYNTHESIS GAS GENERATOR WAS RESULT OF EMBRITTLEMENT DUE TO EXCESSIVE CARBIDE PRECIPITATION AND THERMAL STRESSES EXPERIENCED DURING CYCLING.

RECOMMENDATIONS:
1. REDESIGN BURNER SECTION OF THE GENERATOR SO THAT HIGH TEMP. OF OPERATION CAN BE AVOIDED.
2. USE INCONEL 601 WHICH HAS GOOD RESISTANCE TO CARBURIZATION AND OXIDATION.
3. EXPLORE THE USE OF REFRACTOR MATERIALS AND WATER COOLING OF BURNER COMPONENTS TO PREVENT FUTURE GAS GENERATOR FAILURES.
MET. ANALYSIS OF CORRODED INCOLOY 800 TUBE FROM A RECYCLE GAS HEATER.

THE TUBE HAD A VERY COMPLEX HISTORY. NOT REPRESENTATIVE OF NORMAL OPERATING CONDITIONS.

CONCLUSIONS

1. THE PIPE HAS BEEN DEGRADED BY A COMBINATION OF CARBURIZATION, SULFIDATION, AND OXIDATION.

2. CARBURIZATION IN DEPTH PRECEDED THE OTHER REACTIONS AND ESTABLISHED A Cr-RICH PHASE AT THE GRAIN BOUNDARIES.

3. INCOLOY 800 DOES NOT APPEAR TO HAVE ADEQUATE RESISTANCE TO OXIDATION IN THE ENVIRONMENT FOR THIS APPLICATION. A HIGHER CR ALLOY WHICH CAN FORM A MORE PROTECTIVE OXIDE FILM TO RETARD THE DIFFUSION OF CARBON AND SULFUR TO THE UNDERLYING METAL MAY GIVE BETTER PERFORMANCE. ADDITIONS OF AL, TI, AND SI ALSO WOULD BE HELPFUL.

EXAMINATION OF TYPE 321 S.S. FURNACE TUBE FROM B205 FURNACE

SUMMARY

FAILURE: FURNACE TUBES HAVE EXPERIENCED FREQUENT TROUBLE FROM THINNING. THINNING ORIGINATES FROM INSIDE OF TUBES.

SERVICE LIFE: UNKNOWN

ENVIRONMENT: 900F, 3-4% CO, 26% CO2, 70% N2, 20-75 PPM COS.

MATERIAL: 321 S.S. 1-5/8" OD X 0.109" W.T.

RESULTS

1. INITIALLY GET CARBURIZATION, NITRIDING, AND SOME SULFIDATION THEN GET OXIDATION.

2. SULFUR ATTACK OCCURS, BUT NOT ENOUGH TO BE SIGNIFICANT AT THESE TEMPERATURE.

3. MAJOR METAL LOSS AND THINNING IS DUE TO OXIDATION OF CHROMIUM.

4. THINNING IS FROM INSIDE. OXIDATION ON OUTSIDE IS ALONG GRAIN BOUNDARIES.
I.N.

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CO2

PIPING

LETTER, 11/17/75, FROM J. LETERLE, CONOCO, TO D. GLASER, STEARNS AND ROGER.
HEATER B-205 EXAMINATION AFTER RUN 26A

SUMMARY

DISCUSSION OF EXCESSIVE THINNING OF FURNACE HEATER TUBE DUE TO OXIDATION
AND SULFIDATION. EXPECTED SULFUR LEVEL IS 10-100 PPM AT 900F AS COS. THIS IS
TOO LOW FOR AMOUNT OF DAMAGE SEEN IN TIME EXPOSED. MUST BE UNEXPECTED
SULFUR PRESENT.
RECOMMEND: INSTALL FILTER TRAP TO ANALYSIS SOLID PARTICLES IN FEED STREAM TO
FIND SOURCE OF EXCESS SULFUR.

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I.N.

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EXXON

PIPING

INTRALAB REPORT, 6/30/75, FROM J. SLUSSER TO D. KINZLER, EXXON RESEARCH & ENGR.
ANALYSIS OF COOLING COIL SAMPLES

SUMMARY

FAILURE: HEAVY CORROSION AND METAL LOSS IS OCCURRING ON EXTERIOR SURFACES
OF TUBE ELBOWS. STRAIGHT SECTIONS HAVE MINOR PITTING CORROSION.
SERVICE LIFE: 60 HRS.
ENVIRONMENT: CO2, SO2, O2 AT TEMPERATURES OF 1500-1850 F. TUBESIDE COOLING WATER.
MATERIAL: TUBES ARE 3/4" OD, 0.048" W.T. 316 S.S. ELBOWS ARE 316L S.S.
CAUSE: HIGH TEMPERATURE GAS ATTACK (OXIDATION, SULFIDATION) HAS CAUSED MOST
OF THE CORROSION. PITTING CORROSION POSSIBLY CAUSED BY HIGH TEMPERATURE
SLAG ATTACK. WORST CORROSION AND METAL LOSS OCCURRED ON TUBE ELBOWS DUE TO
CONTINUOUS EROSION OF THE CORROSION PRODUCT RESULTING IN THE CONSTANT
EXPOSURE OF FRESH SURFACES TO THE CORROSIVE MEDIUM.
RECOMMENDATIONS: COOLING WATER SHOULD BE TURNED ON AT STARTUP OF THE REACTION
TO KEEP TUBING TEMPERATURES < 1600 F. IF THIS IS IMPRACTICAL THEN USE
310 S.S. WHICH HAS BETTER HIGH TEMPERATURE CORROSION RESISTANCE.
FAILURE ANALYSIS REPORT, 11/30/73, FROM M. HOWES, IITRI, TO F. SCHRA, IGT.
METALLOGRAPHIC EXAMINATION OF FIVE SAMPLES FROM THE HYGAS PLANT

1. SLURRY DRYER GRID: INCOLOY 800. SERVICE LIFE OF 6 MONTHS. ENVIRONMENT: LIGNITE BED AT 600 F FLUIDIZED WITH A GAS (CONTAINS 13% H2S) AT 1200 F AND 1000 PSI. FAILURE OCCURRED BY CORROSION AT SCREW THREADS. RECOMMEND COATING AND REDESIGN TO ELIMINATE SCREW THREADS.

2. WELDED 4 X 3 IN REDUCER: 446 S.S. SERVICE LIFE OF 6 MONTHS. ENVIRONMENT: 1500 F AT 25 PSI, FLUIDIZED LIGNITE-INTERNAL, NITROGEN-EXTERNAL. FAILURE CAUSED BY CRACK THAT STARTED IN THE WELD BETWEEN THE PIPE AND FLANGE AND PROPAGATED IN BOTH DIRECTIONS. REASON BEING MATERIAL EMBRITTLEMENT AND RESIDUAL STRESSES. RECOMMEND STRESS RELIEVING.

3. BELLows: INCOLOY 800 (20 GAUGE). SERVICE LIFE OF 9 MONTHS. ENVIRONMENT: 1650 F IN A CORROSIVE ATMOSPHERE. FAILURE OCCURRED BY COMBINATION OF SURFACE OXIDATION AND INTERGRANULAR ATTACK. RECOMMEND REDesign.

4. REFORMER QUENCH POT SHELL: INCOLOY 800. SERVICE LIFE OF 6 MONTHS. ENVIRONMENT: 1650 F AT 100 PSI. FAILURE DUE TO THERMAL FATIGUE CAUSED BY FLUCTUATING TEMPERATURE CONDITIONS. RECOMMEND REDesign.

5. BUTTERFLY VALVE: 446 S.S. SERVICE LIFE OF 2 YEARS. ENVIRONMENT: 1400 F. FAILURE DUE TO INTERNAL CORROSION AND RESIDUAL STRESSES. RECOMMEND STRESS RELIEVING.

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REPORT, 6/27/74, FROM W. SALOT, ALLIED CHEMICAL CORP.
TROUBLE WITH HK-40 HIGH-PRESS. REFORMER TUBE OPERATION IN AMMONIA PLANTS

SUMMARY

SUMMARY OF DATA FROM REFORMER INFORMATION NETWORK. NETWORK CONSISTS OF 16 MEMBERS COVERING 21 REFORMERS.
1. TUBE LIFE IMPROVEMENTS HAVE BEEN ACCOMPLISHED WITHOUT COMPLETE TUBE REPLACEMENT.
2. PROJECTED AVERAGE TUBE LIFE HAS INCREASED SUBSTANTIALLY SINCE 1972.
3. SLIGHT INCREASES IN TUBE W.T. OVER 1ST GENERATION REFORMERS NO LONGER APPEARS DETERIMENTAL. AN OPTIMUM THICKNESS MAY EXIST.
4. REPLACEMENT TUBE SPECS COVER A VARIETY OF CHANGES IN THICKNESS, MATERIALS AND OTHER REQUIREMENTS.
5. THESE REFORMERS ARE BEING COMPLETELY RETUBED WHEN MOST OF THEIR TUBES STILL HAVE MANY YEARS REMAINING LIFE.
FAILURE ANALYSIS REPORT, 7/25/75, BY A. MAC NAB, C.F. BRAUN CO.
THERMOCOUPLE SHEATHS—HYGAS

SUMMARY

FAILURE: THERMOCOUPLE TUBES USED IN THE STEAM-OXYGEN GASIFIER WERE BECOMING EMBRITTLED AND CORRODED AFTER SHORT EXPOSURES.
SERVICE LIFE: UNKNOWN
ENVIRONMENT: 2000°F. 0.4% H2S.
MATERIAL: INCONEL 600 AND INCONEL 702.
CAUSE: "GREEN ROT" CORROSION WAS THE PRIMARY MECHANISM. OCCURS IN WEAKLY OXIDIZING ENVIRONMENT. SULFIDATION ALSO OCCURRED AND THIS WAS DUE TO EXPOSURE TO REDUCING SULFIDE CONDITIONS.

RECOMMENDATIONS
1. USE AIR/NITROGEN PURGE.
2. CHANGE MATERIAL TO 310 S.S. OR INCOLOY 800.
3. LOWER NI CONTENT, INCREASE AMOUNT OF Fe AND Cr IN ALLOYS USED.

FAILURE ANALYSIS REPORT, 10/14/75, BY K. NATESAN, ANL.
ANALYSIS OF THERMOCOUPLE FROM HYGAS

SUMMARY

FAILURE ANALYSIS OF THERMOCOUPLE IN STEAM-OXYGEN GASIFIER ENVIRONMENT: 1200 F, 500 PSI STEAM
FAILURE CAUSE: INTERNAL OXIDATION OF CR AND AL CAUSES CRACKING OXIDATION DEPENDS STRONGLY ON CR CONTENT AND TEMPERATURE
RECOMMEND: INCREASE CR TO CREATE PROTECTIVE LAYER OF OXIDE USE 310 S.S.
PITTING
I.N. | PROCESS | SEARCH CRITERIA
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363 | CLEAN COKE | BELLows

QUARTERLY REPORT, 2/77, BY USS ENGINEERS AND CONSULTANTS, INC.
CLEAN COKE PROCESS-REPORT FOR FOURTH QUARTER 1976

SUMMARY

BINDER PREPARATION PDU: A LEAK OCCURRED IN THE 321 S.S. EXPANSION BELLows LOCATED BETWEEN THE MAIN FEED HEATER AND THE CONTINUOUS COLUMN. EXAM. REVEALED PITTING ON THE INTERIOR SURFACE OF THE LEAK AREA. THE OIL THAT FLOWED THROUGH THE BELLows CONTAINED 247 PPM OF CHLORIDE ION AND 0.8% WATER. NORMAL OPERATING TEMPERATURE IS 450 F. THE DESIGN WAS SUCH THAT THE OIL COULD NOT BE COMPLETELY DRAINED FROM THE SYSTEM DURING DOWNTIME. THE CORROSION COULD HAVE OCCURRED DURING THIS PERIOD OR COULD BE DUE TO THE CHLORINE IN THE OIL.

A NEW COIL HAS BEEN FABRICATED OF 316 S.S. AND THE DESIGN MODIFIED TO ALLOW COMPLETE DRAINAGE OF OIL FROM THE SYSTEM DURING DOWNTIME.

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I.N. | PROCESS | SEARCH CRITERIA
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110 | CO2 | BELLows

FAILURE REPORT FROM H. CROWDER TO J. LETERLE, CONOCO, 11/19/74.
FAILURE OF S.S. BELLows FROM LINE CD-206

SUMMARY

FAILURE OF S.S. BELLows FROM LINE CD-206
FAILURE: PITTING CORROSION OF BELLows FROM ACCEPTOR TRANSFER LINE BETWEEN THE REGENERATOR AND THE GASIFIER.
CAUSE: MOIST DEPOSITS ON INNER SURFACE OF BELLows. PRESENCE OF CHLORIDES.
RECOMMENDATIONS: NONE MADE.
FAILURE ANALYSIS REPORT SUMMARY FROM S.T. MCLAURY TO O. GLASER, CONOCO, 12/9/74.
BELLOWS FROM EJ-4 AND EJ-5.

SUMMARY

BELLOWS FROM EJ-4 AND EJ-5.
EJ-4 BELLOWS-FAILED BY SCC-CL.
EJ-5 BELLOWS-FAILED BY PITTING CORROSION ALSO DUE TO CHLORIDES.
SINCE OPERATING CONDITIONS ARE PRESUMED TO BE THE SAME IT IS UNKNOWN WHY CRACKING OCCURRED IN ONE BELLOWS AND PITTING CORROSION OCCURRED IN THE OTHER BELLOWS.

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SEM AND OPTICAL EXAM. REVEALED PITS ON INTERIOR AND EXTERIOR SURFACES IN THE VICINITY OF COMPLETE PERFORATIONS BUT NOT IN AREAS OF APPARENTLY SOUND METAL.
X-RAY ANALYSIS ESTABLISHED THE PRESENCE OF METAL-COAL REACTION PRODUCTS IN CLOSE PROXIMITY TO THE EDGE OF A PERFORATION. IT WILL BE ESTABLISHED WHETHER THIS IS UNIQUE TO PERFORATED AREAS OR HAS BROAD DISTRIBUTION.
BELLOWS FAILURE RESULTING FROM INITIAL DEFECTS (MANUFACTURING) MUST ALSO BE CONSIDERED.
SEE I.N. 186, 194, 210, 274, 285, 286 FOR FURTHER INFORMATION.
I.N. PROCESS SEARCH CRITERIA
286 HYGAS BELLOWS

TELECON, 5/12/76 AND 5/13/76, FROM S. GREENBERG, ANL, TO B. ORCHARD, IGT.
EXPANSION JOINT-GASIFIER TRANSFER LINE.

SUMMARY

MANY HOLES AND PITS DISCOVERED IN INCONEL 800 BELLOWS. DEPOSIT ON INNER SURFACE IS MAINLY THE RESULT OF COUNTER-CURRENT CLEANING OPERATIONS. CORROSION FILM ON EXTERIOR IS RESULT OF CONTAMINATION OF N2 ATMOSPHERE BY IN-LEAKAGE OF COAL, PRODUCT GAS, OIL AND WATER. METALLOGRAPHIC ANALYSIS INDICATES PITTING INITIATED AT INSIDE SURFACE. WATER CONDENSATION DURING SHUTDOWN MAY BE A CONTRIBUTING FACTOR. EVIDENCE FOUND OF SULFIDATION AND OVER-TEMPERATURE OPERATION (1450F). NORMAL OPERATING TEMP. IS 1000F.

MATERIAL RECOMMENDATIONS
1. IF PITTING ATTACK IS CAUSE OF FAILURE, 316 OR 317 S.S. COULD BE USED (USE INVOLVES RISK OF SCC).
2. IF HIGH TEMP. GASEOUS CORROSION IS ALSO IMPORTANT THEN INCONEL 625 OR HASTELLOY C WOULD BE PREFERABLE (ALSO REDUCE RISK OF SCC).

SEE I.N. 186, 194, 210, 236, 274, 285 FOR FURTHER INFORMATION.

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I.N. PROCESS SEARCH CRITERIA
194 HYGAS BELLOWS

TELECON, 6/2/76, FROM S. GREENBERG, ANL, TO B. BAIR, IGT.
EXPANSION JOINT-GASIFIER TRANSFER LINE.

SUMMARY

ANALYSIS OF COAL PRODUCT FOUND IN BELLOWS SHOWED A SOLUBLE CHLORIDE CONC. OF APPROX. 0.2%. THUS PITTING CORROSION HYPOTHESIS IS POSSIBLE.
FUTURE BELLOWS MIGHT BE FABRICATED FROM ALLOYS RESISTANT TO PITTING CORROSION 316 OR 317 S.S., INCONEL 625, HASTELLOY C. HOWEVER, NO EXISTING MATERIAL MIGHT BE SATISFACTORY FOR BOTH AQUEOUS PITTING CORROSION AS WELL AS HIGH TEMP. SULFIDATION IN TERMS OF A LONG SERVICE LIFE.

SEE I.N. 186, 210, 236, 274, 285, 286 FOR FURTHER INFORMATION.
METALLURGICAL ANALYSIS OF INCOLOY 800 TUBE FROM B-201-1A HEATER COIL AFTER RUNS 20-22. FOLLOW-UP TO PREVIOUS LOCKHEED REPORT (LMSC-D407190). THAT REPORT DESCRIBED PITTING IN THE COIL UP TO RUN NO. 19. THE ANALYSIS IN THIS REPORT WAS FROM A SAMPLE TAKEN AFTER RUN NO. 22. 

CONCLUSIONS 
1. PREEXISTING PITS DID NOT GROW IN SIZE AND NEW PITS DID NOT FORM DURING A 510-HR EXPOSURE OF INCOLOY 800 PIPE TO RECYCLE GAS WITH THE ADDITION OF 30-50% STEAM.
2. THERE WAS NO SIGNIFICANT INCREASE IN THE AMOUNT OF INTERNAL CARBURIZATION AND SULFIDATION DURING THIS EXPOSURE.
3. THE ADDITION OF >30% STEAM TO THE RECYCLE GAS COUPLED WITH LITTLE, IF ANY, EXPOSURE TO ALTERNATE REDUCING/OXIDIZING CONDITIONS APPEARS TO BE RESPONSIBLE FOR THE GOOD RESISTANCE TO HIGH-TEMPERATURE PITTING.

FAILURE: HEAVY CORROSION AND METAL LOSS IS OCCURRING ON EXTERIOR SURFACES OF TUBE ELBOWS. STRAIGHT SECTIONS HAVE MINOR PITTING CORROSION. 
SERVICE LIFE: 60 HRS.
ENVIRONMENT: CO2, SO2, O2 AT TEMPERATURES OF 1500-1850 F. TUBESIDE COOLING WATER.
MATERIAL: TUBES ARE 3/4" OD, 0.049" W.T. 316 S.S. ELBOWS ARE 316L S.S.
CAUSE: HIGH TEMPERATURE GAS ATTACK (OXIDATION, SULFIDATION) HAS CAUSED MOST OF THE CORROSION. PITTING CORROSION POSSIBLY CAUSED BY HIGH TEMPERATURE SLAG ATTACK. WORST CORROSION AND METAL LOSS OCCURRED ON TUBE ELBOWS DUE TO CONTINUOUS FRESHENING OF THE CORROSION PRODUCT RESULTING IN THE CONSTANT EXPOSURE OF FRESH SURFACES TO THE CORROSIVE MEDIUM.
RECOMMENDATIONS: COOLING WATER SHOULD BE TURNED ON AT STARTUP OF THE REACTION TO KEEP TUBING TEMPERATURES < 1600 F. IF THIS IS IMPRACTICAL THEN USE 310 S.S. WHICH HAS BETTER HIGH TEMPERATURE CORROSION RESISTANCE.
FAILURE ANALYSIS REPORT, 10/31/74, BY A. MAC NAB, C. F. BRAUN AND CO.
FAILURE OF CARBON STEEL CONDENSER TUBING

SUMMARY

PITTING AND NONUNIFORM CORROSION OF CARBON STEEL PIPE IN HEAT EXCHANGER.
ATTACK IS FROM INSIDE OF TUBING INDICATING AN AGGRESSIVE ENVIRONMENT IN
PROCESS RAW GAS CONDENSATE. PIPE IS 3/4" OD X 0.965" W.T. CARBON STEEL. IN
SERVICE FOR 6 MONTHS GASIFICATION OUT OF THREE YEARS USE. ENVIRONMENT:
PROCESS CONDENSATE IS HIGHLY ACID. AMMONIA ADDED FOR PH CONTROL.
METAL LOSS: 53 MILS MAX, 33 MILS AVG.

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PARTIAL REPORT, NO AUTHOR, NO DATE.
CORROSION OF PDU COMPONENTS—PROJECT LIGNITE

SUMMARY

SUMMARY OF 22 GENERAL SAMPLES SHOWING CORROSION AND/OR EROSION. 16 SAMPLES
SHOWED SCC, 3 SAMPLES SHOWED PITTING ATTACK AND 3 SUFFERED FROM EROSION.
REMEDIAL SOLUTIONS
1. REPLACED 316 S.S. WITH INCOLOY 800 FOR NEW COILS, DISSOLVERS AND FLUID
   LINES.
2. REPLACED CARBON STEEL SEPARATORS WITH CAST 316 S.S.
3. THERMOCOUPLE SHEATHS REPLACED WITH INCONEL 600.
4. EVALUATE NOT FOR CRACK DETECTION.
5. CHECK FOR CAUSATIVE AGENTS OTHER THAN CHLORIDES.
METALLURGICAL ANALYSIS OF STAINLESS STEEL SAMPLES

TYPE 316 S.S. HEATER COILS ARE CRACKING FROM CL-SCC. CRACKS HAVE BEEN MOST SEVERE IN AREAS OF HIGH RESIDUAL STRESS FROM COLD WORKING AND AT POINTS WHERE A CHLORIDE CONCENTRATING MECHANISM SUCH AS FLASHING WATER HAS BEEN OPERATIVE.

HEATER OUTLET (ABOUT 800°F) AND THE HIGH PRESSURE SEPARATOR (ABOUT 550°F).

CHLORIDE SCC HAS OCCURRED WHEREEVER TENSILE STRESSES HAVE BEEN PRESENT. COLD WORK HAS PROVIDED THE CHIEF SOURCE OF TENSILE STRESS, BUT STRESSES FROM WELDING AND, POSSIBLY, OPERATIONAL STRESSES (INTERNAL HOOP STRESS AND BENDING OF PIPING INSTALLATION OR FROM THERMAL EXPANSION) ARE ALSO PRESENT.

CL STRESS CORROSION IN THE TYPE 316 S.S. HEATER TUBE HAS NOT BEEN ACCOMPANIED BY PITTING. ON THE OTHER HAND CRACKS IN TYPE 304 S.S. HAVE ALWAYS STARTED AS PITS. THE ONLY PITTING OCCURRING IN TYPE 316 S.S. WAS IN A SENSITIZED REGION WHICH CONTAINED NO CRACKS.

LOCALIZED CORROSION IN THE FORM OF PITTING AND WASHING AWAY OF A SMALL AREA OF THERMOCOUPLE AND STRAIGHT PIPING TO E-3 WAS THE RESULT OF ACID ATTACK.

LOWERING OF STRESSES BY STRESS-RELIEF AND ALLOY CHANGES ARE THE PRIMARY RECOMMENDATIONS FOR ELIMINATING THE CHLORIDE CRACKING PROBLEM.

APPENDIX B: SUMMARY OF METAL FAILURES DUE TO CORROSION WHICH HAVE OCCURRED IN PROJECT LIGNITE FROM INITIAL START-UP TO 7/24/75.
SUMMARY

PREHEATER COIL FAILURES: E-1 COIL NO.1. COIL WAS CONSTRUCTED OF 95\% OF 9/16" OD X 5/16" ID COLD DRAWN 316 S.S. TUBING WHICH HAD BEEN FABRICATED INTO A HELICAL COIL APPROX. DIAM. OF 9-9/16". COIL FRACTURED AT BOTTOM NEAR SLURRY INLET AFTER ABOUT 400 HRS (125 HRS WITH COAL). TEMP. 750-800°F. PRESS. 1250-1500 PSI. E-1 COIL NO. 2. FRACTURED IN SERVICE AFTER 250 HRS (200 HRS WITH COAL). E-1 COIL NO. 3. FRACTURED IN SERVICE AFTER ONLY 28 HRS (13 HRS WITH COAL). COIL FRACTURED NEAR TOP ABOUT 60" FROM SLURRY INLET. THIS COIL HAD BEEN ANNEALED PRIOR TO FABRICATION.

ANALYSES BY PROJECT LIGNITE STAFF, GULF R&D CO., RALPH PARSONS CO. DETERMINED THAT CAUSE OF FAILURE WAS SCC-CL. REPLACED 316 S.S. COILS WITH INCOLOY 800.

THERMOWELLS: PERFORATIONS OF SHEATHS WITH RESULTANT LEAKAGE LOCATED AT EACH END OF COOLER E-2 IN THE LINE FROM DISSOLVER R-1 TO THE SEPARATOR S-1.

FABRICATED AT PLANT FROM 1/8" OD 304 S.S. PARSONS CO. ANALYSIS SHOWED 1) DEEP PITTING, 2) TRANSGRANULAR SCC AT BOTTOM OF PITS. REPLACED WITH INCONEL 600 ONLY HIGH N ALLOY READILY AVAILABLE IN REQUIRED FORM.

DISSOLVER VESSELS: SLOW LEAK DEVELOPED AT BOTTOM CLOSURE (OUTLET) OF ONE OF THE TWO DISSOLVER VESSELS. CONSTRUCTED OF A 19' 3" LENGTH OF 316 S.S. PIPE WITH A 4-1/2" OD X 3-7/16" ID. ALL PIPING, CLOSURES, ETC. WERE OF 316 S.S. CRACKING OCCURRED ON ALL CLOSURES BUT WAS SEVERS ON OUTLET CLOSURES. ANALYSIS BY PROJECT LIGNITE STAFF REVEALED BRANCHED TRANSGRANULAR SCC. REPLACED MATERIAL WITH INCOLOY 800.

SEVERAL ADDITIONAL EXAMPLES ARE SHOWN OF CORROSION, EROSION, AND SCC MOSTLY IN 316 S.S. COMPONENTS. ALL REPLACED WITH INCOLOY 800. BANNED USE OF TRICHLOROETHANE-HELP TO ELIMINATE CL SINCE MAKING THE ALLOY CHANGES. MANY HUNDREDS OF HOURS OF SERVICE INDICATE NO SCC OR SEVERE PITTING.
REPORT, 2/76, BY M. LAVIGNE, CANADA CENTRE FOR MINERAL AND ENERGY TECHNOLOGY.

SELECTION OF MATERIALS AND FABRICATION METHODS FOR A DEMONSTRATION PLANT PRESSURE VESSEL

SUMMARY

METALLOGRAPHIC EXAMINATION OF 5 SAMPLES CUT FROM THE HOTTEST SECTIONS OF THE ENERGY RESEARCH LABORATORIES 1 BBL/DAY PILOT PLANT.

1. THIRD HEATER TUBE OF FEED LINE TO THE REACTOR VESSELS: 304 S.S. TUBE (APPROX. 3 FT. LONG X 9/16" OD X 5/32" ID) OPERATING TEMP., 450 C. REMOVED AFTER 6 MONTHS OPERATION DUE TO BEING PLUGGED. EXAM REVEALED NO CORROSION. CHROMIUM CARBIDES WERE PRECIPITATED AT GRAIN BOUNDARIES AND ON SLIP PLANES.


3. TOP PLUG OF PRESSURE VESSEL: 1-1/2" OD 316 S.S. SERVICE LIFE OF 2 YEARS. INNER SURFACE OF PLUG WAS PITTED TO A DEPTH OF 0.002".

4. TUBE FROM PRESSURE VESSEL TO HOT RECEIVERS: 304 S.S. TUBE (1/4" OD X 1/8" ID) CARRIED THE PRODUCTS FROM THE PRESSURE VESSEL TO THE HOT RECEIVER AT TEMPERATURES OF 370-460 C. EXAMINATION REVEALED INTERGRANULAR PITTING CORROSION WITH GENERAL CORROSION ALSO OCCURRING.

5. HOT RECEIVER DOWN TUBE: 304 S.S. SAMPLE TAKEN FROM FIRST SECTION OF THE DOWN TUBE WHERE OPERATING TEMPERATURE IS 370 C. EXAMINATION REVEALED PITTING CORROSION ON BOTH OD AND ID OF PIPE.

DESCRIPTION GIVEN OF VARIOUS FABRICATION METHODS FOR ASSEMBLY OF PRESSURE VESSELS.

THE RESULT OF THIS INVESTIGATION WAS THE SELECTION OF 316 S.S. FOR USE IN THE PRESSURE VESSEL AND RELATED COMPONENTS.

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REPORT, 1/26/77, BY R. COOPER, ORNL.
CORROSION ANALYSIS OF BOILER TUBES FROM A 100 HP BOILER

SUMMARY

ANALYSIS: CORROSION ANALYSIS WAS PERFORMED ON A178 BOILER TUBE SEGMENTS REMOVED FROM A 100 HP BOILER OPERATED BY PERC.
SERVICE LIFE: 1000 HRS
ENVIRONMENT: 340°F STEAM AND FLUE GAS GENERATED FROM THE COMBUSTION OF NO 6 FUEL OIL-COAL SLURRY.
MATERIAL: CARBON STEEL, ASTM A178 SPEC.
DISCUSSION: STEAM SIDE CORROSION CAUSED LOCALIZED AREAS OF PITTING AND HEAVY SCALE ON PIPE EXTERIORS. THIS MAY RESULT FROM EITHER EXPOSURE TO ATMOSPHERIC CONDITIONS DURING BOILER SHUTDOWN AND DISASSEMBLY OR AN INCREASED DISSOLVED OXYGEN CONTENT OF THE BOILER WATER DUE TO IMPROPER PROCESS CONTROL.
FLUE GAS CORROSION RESULTED IN A 3 MIL SCALE FORMING ON THE INTERIOR WALLS OF THE PIPES. A LARGE PART OF THE SCALE OBSERVED ON THE TUBE WALLS COULD BE THE RESULT OF EXPOSURE TO AN ACIDIC CONDENSATE FORMED DURING THE MANY SHUTDOWN CYCLES. SOME OF THE SCALE MAY HAVE FORMED AS A RESULT OF EXPOSURE OF THE TUBES TO ATMOSPHERIC CONDITIONS FOLLOWING REMOVAL FROM THE FURNACE.

CONCLUSIONS

1. ANALYSIS REVEALED A MEASURABLE CORROSION SCALE ON THESE TUBES.
2. WHAT FRACTION OF THIS CORROSION IS THE RESULT OF THE FLUE GAS ENVIRONMENT IS UNKNOWN.
3. ADDITIONAL ANALYSIS OF THESE TUBE SEGMENTS WILL NOT PROVIDE USEFUL DATA.
4. SINCE THERE IS NO PRACTICAL METHOD OF UTILIZING SURVEILLANCE COUPONS, THE DESTRUCTIVE ANALYSIS OF BOILER TUBES IS THE ONLY AVAILABLE METHOD FOR MEASURING CORROSION.
5. THESE MEASUREMENTS WILL ONLY PRODUCE MEASUREMENTS OF SCALE THICKNESS AND NOT PRECISE METAL LOSS DATA.

RECOMMENDATIONS

1. SURVEILLANCE TUBES SHOULD INITIALLY BE CORROSION FREE.
2. AS RECEIVED MATERIAL SHOULD BE SEALED IN AN INERT ATMOSPHERE AND HELD FOR REFERENCE.
3. AN EFFORT SHOULD BE MADE TO MINIMIZE THE NUMBER OF SHUTDOWN AND START-UP CYCLES.
4. SAMPLE TUBES SHOULD BE EXPOSED FOR A MINIMUM OF 1000 HRS.
5. AT END OF RUN, TUBES SHOULD BE PROMPTLY REMOVED FROM THE BOILER, SECTIONED INTO SAMPLES, SEALED IN AN INERT ATMOSPHERE, AND SENT TO THE TESTING LAB.

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### Fail Analysis RPT, 4/30/76, from L. Oden, Albany Met. Res. CTR., to F. Gromicko, PERC.

**EVALUATION OF CRACKED 316 S.S. PIPE SECTION FROM SYNTHANE PILOT PLANT**

**SUMMARY**

The failed preheater pipe (from the gas disengagement zone in the gas entrained feed system) shows general corrosion and pitting of the inner surface in addition to severe cracking which is probably SCC. The cracking is both inter- and transgranular.

The pipe is welded and the weld metal was attacked less severely than the parent metal. No sensitization of metal adjacent to weld was observed, or cracking of weld metal.

Electron microprobe analysis of scale shows sulfur and chlorine present in proportions of 5-10%.

316 S.S. is susceptible to chloride ions. Quantity of chlorine in scale indicates a continuous supply from the gas stream. Sulfur accentuates corrosion by decreasing the protective nature of the oxide scale. Recommend using carbon drum to remove impurities from the gas.

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### Telecon, 3/31/77, from S. Danyuk, ANL, to D. Duris, SYNTHANE

**BALLOONING OF C-MO STEEL PIPE AND PITTING IN CYCLONE DIPLEG**

**SUMMARY**

**A. BALLOONING OF C-MO STEEL PIPE**

1. Design temp. and pressures could not provide sufficient stress to plastically deform the pipe.
2. Wall thickness measurements seem reasonable.
3. Tensile tests, hardness tests, chemical analysis and metallographic analysis will be performed.
4. At present cause of ballooning is unknown.

**B. CYCLONE DIPLEG-Incoloy 800**

1. Corrosion pits have developed.
2. Sections of pipe are to be shipped to ANL.
3. Synthane requests material identification and reason for pitting.

See I.N. 356, 357 for further information on ballooning problem.
FAILURE: PITS, APPROX. 1/16" DEEP, HAVE BEEN SEEN IN GASIFIER THERMOWELLS.
SERVICE LIFE: APPROX. 500 HRS.
ENVIRONMENT: TEMP. 1500-1600 F.
MATERIAL: HASTELLOY C-276.
CAUSE: UNKNOWN. MATERIAL IS SUPPOSED TO HAVE GOOD PIT RESISTANCE.
ACTION: A SECTION OF THERMOWELL IS BEING SENT TO ARGONNE FOR ANALYSIS.
FAILURE ANALYSIS REPORT, N.D., BY J. FISCHER, R. DE ANGELIS, O. HAHN, P. GILLIS, U. OF KY.
METALLOGRAPHIC ANALYSIS OF A COAL GASIFIER PLATE FAILURE

SUMMARY

FAILURE: CRACK PROPAGATION THROUGH THE WALL NEAR THE GRATE WHICH ALLOWED COOLANT TO FLOW INTO THE REACTION CHAMBER.
ENVIRONMENT: OPERATED AT A NOMINAL PRESSURE OF 120 PSI BUT WAS DESIGNED TO BE CAPABLE OF OPERATING AT 350 PSI.
CONCLUSIONS: IT IS TENTATIVELY CONCLUDED THAT THE CRACK WHICH LED TO FAILURE DID SO ONLY BECAUSE IT WAS ABLE TO PROPAGATE THROUGH THE WALL FOLLOWING A PATH ENTIRELY WITHIN THE WELD FILLER METAL. IF THIS IS TRUE THEN THE S.S. LUGS SHOULD BE LOCATED AWAY FROM WELDS WHICH EXTEND THROUGH THE THICKNESS OF THE VESSEL.

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METALLOGRAPHIC EXAMINATION OF 5 SAMPLES CUT FROM THE HOTTEST SECTIONS OF
THE ENERGY RESEARCH LABORATORIES 1 BBL/DAY PILOT PLANT.
1. THIRD HEATER TUBE OF FEED LINE TO THE REACTOR VESSELS: 304 S.S. TUBE
   (APPROX. 3 FT. LONG X 9/16" OD X 5/32" ID) OPERATING TEMP. 450 C.
   REMOVED AFTER 6 MONTHS OPERATION DUE TO BEING PLUGGED. EXAM REVEALED NO
   CORROSION. CHROMIUM CARBIDES WERE PRECIPITATED AT GRAIN BOUNDARIES AND
   ON SLIP PLANES.
2. CENTRAL THERMOCOUPLE WELL OF THE VESSEL: 304 S.S. TUBE (APPROX. 12.5 FT
   LONG X 1/2" OD X 1/4" ID). END SEALED BY WELDING. THE WALL OF THE TUBE
   HAD NO CORROSION. SOME CORROSION PRODUCTS WERE DEPOSITED ON A SECTION
   OF THE WELD.
3. TOP PLUG OF PRESSURE VESSEL: 1-1/2" OD 316 S.S. SERVICE LIFE OF 2 YEARS.
   INNER SURFACE OF PLUG WAS PITTED TO A DEPTH OF 0.002".
4. TUBE FROM PRESSURE VESSEL TO HOT RECEIVERS: 304 S.S. TUBE (1/4" OD X
   1/8" ID) CARRIED THE PRODUCTS FROM THE PRESSURE VESSEL TO THE HOT
   RECEIVER AT TEMPERATURES OF 370-460 C. EXAMINATION REVEALED INTERGRANULAR
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   DOWN TUBE WHERE OPERATING TEMPERATURE IS 370 C. EXAMINATION REVEALED
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VESSELS.

THE RESULT OF THIS INVESTIGATION WAS THE SELECTION OF 316 S.S. FOR USE IN
THE PRESSURE VESSEL AND RELATED COMPONENTS.
INTEROFFICE REPORT, 12/13/74, FROM C. ACKERMAN & L. FURLER TO G. CHENOOWETH, SRC.
INSPECTION OF DISSOLVER A

SUMMARY

TWO SEPARATE LEAKS DEVELOPED IN SLURRY DISSOLVER A (02075001). ON NEXT SCHEDULED SHUTDOWN THE VESSEL WAS CLEANED BY A COMBINATION OF STEAMING, WATER, INERT GAS AND CARBONATE SOLUTION. HEAVY CARBONACEOUS DEPOSITS WERE REMOVED FROM INSIDE THE VESSEL. CAREFUL VISUAL INSPECTION AND ULTRASONIC TESTING TO DETECT CORROSION AND FLAWS WAS CONDUCTED—NO CORROSION OF FLAWS WERE FOUND. OPERATION WAS RESUMED AS NORMAL AND SUBSEQUENT UT AND VISUAL INSPECTION WAS TO BE DONE.

RECOMMEND: DETAILED DIAGNOSTIC FAILURE ANALYSIS

EFFECT: DID NOT REQUIRE SHUTDOWN COULD WAIT TO END OF RUN AND THEN INSPECT.

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INTERNAL REPORT, 9/9/76, FROM C. ACKERMAN & D. CANFIELD TO N. GREENLEE, FT. LEWIS.

INSPECTION AND CLEANING OF 502 PRESSURE VESSELS

SUMMARY

INSPECTION RESULTS

1. Cracks in the cladding inside the cover of the I.P. and recycle condensate separator will require monitoring every 3 or 4 months by ultrasonic inspection when cold. The I.P. separator cover will need to be replaced within the next year.

2. A large crack in the breather ring on the middle outlet nozzle in dissolver "A" covers over 90 degrees of the inner weld. The base metal under the crack has suffered some pitting corrosion.

3. A smaller crack was found in the breather ring in the dissolver "A" inlet nozzle.

4. Chloride initiated cracks were found in one breather ring in a separator but other places had none.

5. Corrosion rack installed 12/10/74 in recycle condensate separator removed by S. IBARRA, HARMARVILLE, for evaluation.

RECOMMENDATIONS

1. The I.P. separator cover be replaced with a new cover with stabilized S.S. cladding or weld overlay.

2. The present I.P. separator cover, recycle condensate separator cover, and dissolver "A" inlet and side nozzles be monitored by ultrasonic inspection about every 3 months.

3. More corrosion samples (including stressed samples) be installed in dissolver "A" below the center outlet, and in each of the separators in the gas phase.

4. Better procedures and equipment for inspection are needed.

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Inspection of SRC 02 Pressure Vessels

Summary

Conclusions and Recommendations

1. The cracking in the top head cladding of the I.P. separator was due principally to polythionic acid SCC. Some evidence of SCC-CL was found in the cladding as well as the 304 S.S. nozzle breather ring.

2. Cracking due to polythionic acid SCC was seen in the recycle condensate separator. Cracking was noted in the cladded top head. No SCC-CL seen.

3. Inspection of nozzles in "A" dissolver revealed that the side nozzle sleeve had separated from the weld which connected it to the nozzle breather ring. The bottom nozzle also had an indication of a rupture in the lining. Evidence of a small crack between the nozzle sleeve and the nozzle breather ring. No significant corrosion of the base metal of the nozzles or the shell.

4. Recommended that all components be returned to service. Periodic ultrasonic monitoring advised. A thorough examination should be conducted in about one year.
PROCESS SEARCH CRITERIA

59 SYNTHANE VALVES

ERDA MAT. AND COMP. FAIL. REPORT, 12/12/75, FROM R. JACKSON, SYNTHANE. BALL UNIT OF XCV-27, EBV BALL VALVE

SUMMARY

FAILURE: EXCESSIVE SCALING AND PITTING OF BALL AND JOURNAL OF VALVE. SERVICE LIFE: INSTALLED BUT HAD SEEN NO SERVICE. ENVIRONMENT: AMBIENT TEMP. MATERIAL: 440C S.S. ACTION: REPLACED PART. ANALYSIS BY COMPANY LAB.

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I.N. PROCESS SEARCH CRITERIA

60 SYNTHANE VALVES

ERDA MAT. AND COMP. FAIL. REPORT, 12/18/75, FROM R. JACKSON, SYNTHANE. BALL UNIT OF XCV-30, EBV BALL VALVE

SUMMARY

FAILURE: ONE SIDE OF BALL HAD PITS IN THE AREA OF THE SEAT. SERVICE LIFE: INSTALLED BUT NOT PLACED IN SERVICE. MATERIAL: 440C S.S. FORGED BAR WITH STELLITE SEAT. ACTION: UNKNOWN.
SULFIDATION
I.N. PROCESS SEARCH CRITERIA
17 CO2 PIPING

LETTER REPORT, 2/16/70, FROM J. LETTERLE, CONOCO.
HIGH TEMPERATURE CORROSION OF PARTS FROM CO2 PILOT PLANT

SUMMARY


CONCLUSIONS
1. IT APPEARS THAT SULFUR HAS REACTED WITH ALL THREE METALS TO FORM A LOW MELTING POINT EUTECTIC OF SULFUR, CHROMIUM, NICKEL, AND IRON.
2. THE 310 S.S. HAS BEEN HIGHLY CARBURIZED.
3. IF THE ENVIRONMENT THAT CAUSED THIS CORROSION IS TYPICAL THEN CAREFUL CONSIDERATION MUST BE GIVEN TO ALLOY SELECTIONS.

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I.N. PROCESS SEARCH CRITERIA
124 CO2 PIPING

INTEROFFICE REPORT FROM H. CROWDER TO F. J. RADD, CONOCO, 3/25/70.
METALLURGICAL STUDY OF REACTOR AND GASIFIER VESSELS FROM THE LIGNITE GASIFICATION PRE-PILOT PLANT, CONCOL

SUMMARY

THE BENCH SCALE LIGNITE GASIFICATION PLANT, OPERATED AT LIBRARY, PA HAS EXPERIENCED HIGH TEMPERATURE CORROSION AND EMBRITTLEMENT PROBLEMS. THIS REPORT SUMMARIZES WORK ON THE FOLLOWING PARTS:
1. GASIFIER VESSEL
2. REGENERATOR VESSEL
3. REGENERATOR PRE-HEAT COIL
4. REGENERATOR THERMOWELL

CONCLUSIONS: ALL FOUR OF THE PIECES HAVE EXPERIENCED ONE OR MORE OF THE FOLLOWING PROBLEMS: HIGH TEMPERATURE SULFUR CORROSION, EMBRITTLEMENT FROM CARBURIZING, EMBRITTLEMENT FROM NITRIDING.
THESE PROBLEMS COVER THE TEMPERATURE RANGE OF 950-1900 F.
INTEROFFICE REPORT, 8/7/72, FROM H. CROWDER TO J. LETERLE, CONOCO.
METALLOGRAPHIC STUDY OF 4-INCH DIAMETER INCOLOY 300 PIPE

SUMMARY

TWO SAMPLES OF REMAINING PIPE (SEE I.N. 88) WERE EXAMINED TO DETERMINE SUITABILITY FOR FURTHER USE.

CONCLUSIONS
1. THE INSIDE SURFACE HAS BEEN DAMAGED BY SULFUR CORROSION AND BY GRAIN BOUNDARY PENETRATION BY SULFUR.
2. OVERHEATING CAUSED SOME INCREASE IN GRAIN SIZE.
3. MOST OF THE WALL THICKNESS IS STILL SOUND ENOUGH TO ALLOW THIS TOP SECTION TO BE USED FOR FURTHER SERVICE.

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FAILURE REPORT, 2/7/74, BY R. PERKINS AND W. COONS, LOCKHEED LAB TO CONSOL.
MET. ANALYSIS OF CORRODED INCOLOY 800 TUBE FROM A RECYCLE GAS HEATER.

SUMMARY

MET. ANALYSIS OF CORRODED INCOLOY 800 TUBE FROM A RECYCLE GAS HEATER. THE TUBE HAD A VERY COMPLEX HISTORY. NOT REPRESENTATIVE OF NORMAL OPERATING CONDITIONS.

CONCLUSIONS
1. THE PIPE HAS BEEN DEGRADED BY A COMBINATION OF CARBURIZATION, SULFIDATION, AND OXIDATION.
2. CARBURIZATION IN DEPTH PRECEDED THE OTHER REACTIONS AND ESTABLISHED A CR-RICH PHASE AT THE GRAIN BOUNDARIES.
3. INCOLOY 800 DOES NOT APPEAR TO HAVE ADEQUATE RESISTANCE TO OXIDATION IN THE ENVIRONMENT FOR THIS APPLICATION. A HIGHER CR ALLOY WHICH CAN FORM A MORE PROTECTIVE OXIDE FILM TO RETARD THE DIFFUSION OF CARBON AND SULFUR TO THE UNDERLYING METAL MAY GIVE BETTER PERFORMANCE.
ADDITIONS OF AL, TI, AND SI ALSO WOULD BE HELPFUL.
FAILURE OF THE GASIFIER PROBE AND SENSOR TUBES

SUMMARY

FAILURE OF THE GASIFIER PROBE AND SENSOR TUBES.
Both the probe and sensor tubes failed as a result of stress enhanced intergranular sulfur corrosion. Probe was 310 S.S. Sensor tubes were 316 S.S. Operating temp. -300-1600F. Purge gas claimed to be inert-suspected, however, of containing a sulfur corrosive. No proof.

Recommendations: Remove stress concentrations produced by differences in thermal expansion and contraction between the insulation basket and the gasifier probe. Remove sulfur from incoming purge gas to prevent premature failure due to high temperature sulfur corrosion.

CONCLUSIONS

1. The bond between the coating and the base metal appears to have been satisfactory.
2. Some of the hard surfacing failed because it is a brittle material which has been damaged by mechanical or thermal shock.
3. Some of the material failed because of high temperature sulfur corrosion.
4. Unless there is a material available that is better than 310 S.S., we suggest that an alonized 310 S.S. thermowell be tried.

DISBONDING OF HARD FACING MATERIAL FROM STAINLESS STEEL THERMOWELLS

SUMMARY

Experimental thermowells were made by applying hard facing materials to 310 S.S. tubes by a plasma-arc welding process. The thermowells were installed in the regenerator, D-203, of the lignite gasification plant.

The thermowells failed after a service life of 24 hrs or less. Chromium carbide hard surfacing material was applied to one thermowell because it had given good service in other severe environments. Aluminum oxide and titanium carbide were applied to two other thermowells to provide additional materials for testing.

CONCLUSIONS

1. The bond between the coating and the base metal appears to have been satisfactory.
2. Some of the hard surfacing failed because it is a brittle material which has been damaged by mechanical or thermal shock.
3. Some of the material failed because of high temperature sulfur corrosion.
4. Unless there is a material available that is better than 310 S.S., we suggest that an alonized 310 S.S. thermowell be tried.
FINAL REPORT FROM D. QUALLS TO J. LEITERLE, CONOCO, 11/7/74.
EVALUATION OF HEATER TUBES FROM RAPID CITY CONSOL

SUMMARY
EVALUATION OF HEATER TUBES FROM RAPID CITY CONSOL
FIVE HEATER TUBE SAMPLES WERE SUBMITTED. THEY HAD BEEN INSPECTED BY MAGNETIC INSPECTION WHICH INDICATED SOME DAMAGE.
A MICROSTRUCTURAL EVALUATION WAS REQUESTED TO CORRELATE ACTUAL DAMAGE WITH MAGNETIC INSPECTION RESULTS.
ALONIZED 304 S.S. TUBE WAS GOOD. THE FOUR SAMPLES OF INCOLOY 800 TUBING CONTAINED VARYING DEGREES OF CORROSION AND STRUCTURAL DAMAGE. THE DAMAGE WAS < 12.2% OF THE MINIMUM MEASURED WALL THICKNESS.

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ERDA MAT. AND COMP. FAIL. REPORT, 8/8/75, FROM C. SCHULZ, CO2.
INLET OF LINE CD-206 IN THE REGENERATOR

SUMMARY
INLET OF LINE CD-206 IN THE REGENERATOR.
FAILURE: THE INNER LINER OF CD-206 BROKE OFF AND FELL DOWN INTO VESSEL.
SERVICE LIFE: 850 HRS
ENVIRONMENT: 1830F FLUE GAS. THE TUBE IS IMMERSED IN A FLUIDIZING BED WITH A SUPERFICIAL VELOCITY OF ABOUT 3 FPS. PARTICLES ARE 6 X 9 MESH DOLOMITE.
MATERIAL OF CONSTRUCTION: 6" X .25" W.T. 310 S.S. WELDED TUBE.
CAUSE OF FAILURE: CORROSION DUE TO SULFUR. THE STRESS AND STRAINS FROM THE FLUIDIZED BED AND THERMAL CYCLING ACCENTUATED THE CORROSION AND WAS THE IMMEDIATE CAUSE OF THE FAILURE.
NO RECOMMENDATIONS MADE.
FAILURE: A 9-FT 9-IN SECTION OF OLD MATERIAL IN THE "A" PASS WAS REPLACED DUE TO THINNING AND A SMALL PIN HOLE.

SERVICE LIFE: 581 HRS SINCE REBUILD.

PRIOR REPAIRS: COIL WAS REBUILT REPLACING ALL THIN SECTIONS IN DEC. 1974 WITH ALONIZED RA-330.

ENVIRONMENT: TEMP. 1000-1500°F. INERT GAS (83-87% N₂, 6-12% CO₂, 0.2-9.6% O₂) AND RECYCLE GAS (80% N₂, 17% CO₂, 3% CO, CARBONYL SULFIDE 50 PPM).

MATERIALS OF CONSTRUCTION: INCOLOY 800 WITH SECTIONS OF ALONIZED RA-330.

CAUSE OF FAILURE: PROBABLY DUE TO CARBURIZATION AND SULFIDATION.

ACTION: DIAGNOSTIC ANALYSIS TO BE PERFORMED BY CONOCO.

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FAILURE: FURNACE TUBES HAVE EXPERIENCED FREQUENT TROUBLE FROM THINNING.

THINNING ORIGINATES FROM INSIDE OF TUBES.

SERVICE LIFE: UNKNOWN

ENVIRONMENT: 900°F. 3-4% CO, 26% CO₂, 70% N₂, 20-75 PPM COS.

MATERIAL: 321 S.S. 1-5/8" OD X 0.109" W.T.

RESULTS
1. INITIALLY GET CARBURIZATION, NITRIDING, AND SOME SULFIDATION THEN GET OXIDATION.
2. SULFUR ATTACK OCCURS, BUT NOT ENOUGH TO BE SIGNIFICANT AT THESE TEMPERATURE.
3. MAJOR METAL LOSS AND THINNING IS DUE TO OXIDATION OF CHROMIUM.
4. THINNING IS FROM INSIDE. OXIDATION ON OUTSIDE IS ALONG GRAIN BOUNDARIES.
FAILURE: THE TUBE WALLS IN SEVERAL AREAS HAD GREATLY THINNED REQUIRING REPLACEMENT OF THE COIL.

SERVICE LIFE: 919 HRS SINCE REBUILDING

ENVIRONMENT: SEE ATTACHMENT FOR HRS AND TYPES OF ATMOSPHERE

PRIOR REPAIRS: COIL WAS REBUILT REPLACING ALL THIN SECTIONS, 12/74. WITH ALONIZED RA 330 AND A 9'-9" SECTION WAS REPLACED 9/75.

MATERIALS: INCOLOY 800 AND RA 330

CONCLUSIONS: THINNING WAS PROBABLY DUE TO A COMBINATION OF CARBURIZATION AND SULFIDATION. NEW COIL FABRICATED OUT OF INCONEL 702.

ACTION: REPLACED PART. ANALYSIS BY COMPANY LABS.

DISCUSSION OF EXCESSIVE THINNING OF FURNACE HEATER TUBE DUE TO OXIDATION AND SULFIDATION. EXPECTED SULFUR LEVEL IS 10-100 PPM AT 900F AS COS. THIS IS TOO LOW FOR AMOUNT OF DAMAGE SEEN IN TIME EXPOSED. MUST BE UNEXPECTED SULFUR PRESENT.

RECOMMEND: INSTALL FILTER TRAP TO ANALYSIS SOLID PARTICLES IN FEED STREAM TO FIND SOURCE OF EXCESS SULFUR.
ERDA MAT. AND COMP. FAIL REPORT, 11/24/75. CO2. C. SCHULZ.
B-205 GAMMA FURNACE COIL. N

SUMMARY

FAILURE: THREE HOLES IN THREE DIFFERENT TUBE PASSES NEAR THE OUTLET END.
SERVICE LIFE: 61.5 HRS
ENVIRONMENT: SEE ATTACHMENT
MATERIAL: INCONEL 702
CONCLUSIONS: FAILURE PROBABLY DUE TO VERY SEVERE CORROSION FROM SULFUR
AND/OR CARBONIZATION.
ACTION: PART REPLACED. ANALYSIS BY CONOCO LABS.

INTRALAB REPORT, 6/30/75, FROM J. SLUSSER TO D. KINZLER, EXXON RESEARCH & ENGR.
ANALYSIS OF COOLING COIL SAMPLES

SUMMARY

FAILURE: HEAVY CORROSION AND METAL LOSS IS OCCURRING ON EXTERIOR SURFACES
OF TUBE ELBOWS. STRAIGHT SECTIONS HAVE MINOR PITTING CORROSION.
SERVICE LIFE: 60 HRS.
ENVIRONMENT: CO2, SO2, O2 AT TEMPERATURES OF 1500-1850 F. TUBESIDE COOLING WATER.
MATERIAL: TUBES ARE 3/4" OD, 0.048" W.T. 316 S.S. ELBOWS ARE 316L S.S.
CAUSE: HIGH TEMPERATURE GAS ATTACK (OXIDATION, SULFIDATION) HAS CAUSED MOST
OF THE CORROSION. PITTING CORROSION POSSIBLY CAUSED BY HIGH TEMPERATURE
SLAG ATTACK. WORST CORROSION AND METAL LOSS OCCURRED ON TUBE ELBOWS DUE TO
CONTINUOUS EROSION OF THE CORROSION PRODUCT RESULTING IN THE CONSTANT
EXPOSURE OF FRESH SURFACES TO THE CORROSIVE MEDIUM.
RECOMMENDATIONS: COOLING WATER SHOULD BE TURNED ON AT STARTUP OF THE REACTION
TO KEEP TUBING TEMPERATURES < 1600 F. IF THIS IS IMPractical THEN USE
310 S.S. WHICH HAS BETTER HIGH TEMPERATURE CORROSION RESISTANCE.
FAILURE ANALYSIS REPORT, 11/30/73, FROM M. HOWES, IITRI, TO F. SCHORA, IGT.
METALLOGRAPHIC EXAMINATION OF FIVE SAMPLES FROM THE HYGAS PLANT

SUMMARY

1. SLURRY DRYER GRID: INCOLOY 800. SERVICE LIFE OF 6 MONTHS. ENVIRONMENT, LIGNITE BED AT 600 F FLUIDIZED WITH A GAS (CONTAINS 1% H2S) AT 1200 F AND 1000 PSI. FAILURE OCCURRED BY CORROSION AT SCREW THREADS. RECOMMEND COATING AND REDESIGN TO ELIMINATE SCREW THREADS.

2. WELDED 4 X 3 REDUCER: 446 S.S. SERVICE LIFE OF 6 MONTHS. ENVIRONMENT, 1500 F AT 25 PSI. FLUIDIZED LIGNITE—INTERNAL, NITROGEN—EXTERNAL. FAILURE CAUSED BY CRACK THAT STARTED IN THE WELD BETWEEN THE PIPE AND FLANGE AND PROPAGATED IN BOTH DIRECTIONS. REASON BEING MATERIAL EMBRITTLEMENT AND RESIDUAL STRESSES. RECOMMEND STRESS RELIEVING.

3. BELLOW: INCOLOY 800 (20 GAUGE). SERVICE LIFE OF 9 MONTHS. ENVIRONMENT, 1650 F IN A CORROSIVE ATMOSPHERE. FAILURE OCCURRED BY COMBINATION OF SURFACE OXIDATION AND INTERGRANULAR ATTACK. RECOMMEND REDESIGN.

4. REFORMER QUENCH POT SHELL: INCOLOY 800. SERVICE LIFE OF 6 MONTHS. ENVIRONMENT, 1650 F AT 100 PSI. FAILURE DUE TO THERMAL FATIGUE CAUSED BY FLUCTUATING TEMPERATURE CONDITIONS. RECOMMEND REDESIGN.

5. BUTTERFLY VALVE: 446 S.S. SERVICE LIFE OF 2 YEARS. ENVIRONMENT, 1400 F. FAILURE DUE TO INTERNAL CORROSION AND RESIDUAL STRESSES. RECOMMEND STRESS RELIEVING.

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DISCUSSION OF MATERIALS TO RESIST SULFIDATION IN PIPING TO THE REACTOR AND SECOND STAGE REDUCER SPARGE RING. ATMOSPHERE WILL BE REDUCING GAS CONTAINING 0.1 MOLE PERCENT H2S AT 2000F. INCOLOY 800 AND 310 S.S. WILL NOT HOLD UP. RECOMMEND 50CR-50NI (ASTM A562 OR UNILOY 50/50) OR ALONIZED 310 S.S. OR ALONIZED INCOLOY 900. 50CR-50NI HAS GOOD SULFIDATION RESISTANCE BUT POOR HIGH TEMP. STRENGTH. RECOMMEND USE OF THIS MATERIAL AS CLADDING TO A SUBSTRATE OF INCOLOY 800 OR 310 S.S.
TARMAN COMMENTS ON MAC NAB'S SUGGESTION OF USING 50CR-50NI AS CLADDING ON INCOLOY 800 AND HIS RECOMMENDATION THAT IGT CONTACT HUNTINGTON ALLOYS FOR INFORMATION.

MEMO FROM BACHTA GIVES INFORMATION FROM HUNTINGTON ALLOYS. PROBLEMS IN GETTING THIS MATERIAL WOULD BE 1) SIZE, BIGGEST PRODUCED BY HUNTINGTON IS 2-1/2" OD, IGT NEEDS 4" OD. 2) CLADDING THE INTERIOR OF THE PIPE. MOST PRODUCTS CLAD ON OUTSIDE ONLY.

INCOCLAD 671/800H PIPE HAS AN INCOLOY 800 SUBSTRATE WHICH PROVIDES STRENGTH AT HIGH TEMPERATURES AND INCONEL 671 (APPROX. 50CR-50NI) CLADDING WHICH PROVIDES RESISTANCE TO SULFIDATION.

1. THERMOCOUPLE PROTECTION TUBE TC-5 REMOVED AFTER RUN 59 IDENTIFIED AS INCOLOY 800.
2. SCALE REMOVED FROM THE OUTSIDE SURFACE OF PRESSURE TAP FI983 AFTER RUN 60 HAD A SULFUR CONCENTRATION IN THE RANGE OF 5-10%.
3. BAIR'S ASSUMPTION OF SULFUR ATTACK APPEARS TO BE JUSTIFIED & RECOMMENDATION OF 310 OR 446 S.S. AS REPLACEMENT SHOULD WORK.
I.N. | PROCESS | SEARCH CRITERIA
--- | --- | ---
213 | HYGAS | PIPING

TELECON, 4/13/76, FROM S. GREENBERG, ANL, TO B. ORCHARD, IGT.
THERMOCOUPLE PROTECTION TUBES, PRESSURE TAP TUBES, GASIFIER TRANSFER LINE BELLows.

SUMMARY

THERMOCOUPLE PROTECTION TUBES: TUBES TC-23 AND TC-5 IDENTIFIED AS INCOLOY 800.
PRESSURE TAP TUBES: SCALE REMOVED FROM OUTER SURFACE CONTAINED 14.4% S.
EXPANSION JOINT BELLows: COAL PRODUCT REMOVED FROM BELLows HAS A SULFUR CONTENT OF APPRX. 3%, VERY CLOSE TO THAT OF FEED MATERIAL.
THESE RESULTS TEND TO CONFIRM VIEW THAT TUBES FAILED AS A RESULT OF SULFUR ATTACK. METALLOGRAPHIC EXAM. IS IN PROGRESS.

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MANY HOLES AND PITS DISCOVERED IN INCOLOY 800 BELLows. DEPOSIT ON INNER SURFACE IS MAINLY THE RESULT OF COUNTER-CURRENT CLEANING OPERATIONS.
CORROSION FILM ON EXTERIOR IS RESULT OF CONTAMINATION OF N2 ATMOSPHERE BY IN-LEAKAGE OF COAL, PRODUCT GAS, OIL AND WATER. METALLOGRAPHIC ANALYSIS INDICATES PITTING INITIATED AT INSIDE SURFACE. WATER CONDENSATION DURING SHUTDOWN MAY BE A CONTRIBUTING FACTOR. EVIDENCE FOUND OF SULFIDATION AND OVER-TEMPERATURE OPERATION (1450F). NORMAL OPERATING TEMP. IS 1000F.

MATERIAL RECOMMENDATIONS
1. IF PITTING ATTACK IS CAUSE OF FAILURE, 316 OR 317 S.S. COULD BE USED (USE INVOLVES RISK OF SCC).
2. IF HIGH TEMP. GASEOUS CORROSION IS ALSO IMPORTANT THEN INCONEL 625 OR HASTELLOY C WOULD BE PREFERABLE (ALSO REDUCE RISK OF SCC).

SEE I.N. 186, 194, 210, 236, 274, 285 FOR FURTHER INFORMATION.
I.N. | PROCESS | SEARCH CRITERIA
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282 | HYGAS | PIPING

TELECON, 6/24/76, FROM S. GREENBERG, ANL, TO B. ORCHARD, IGT.
AAG INTERNAL CYCLONE DIPLEG
MAIN GASIFIER TRANSFER LINE EXPANSION JOINT.

SUMMARY
1) DIPLEG: EXAM. INDICATES CARBURIZATION AND SULFIDATION HAS OCCURRED.
2) GASIFIER TRANSFER PIPING: INTERGRANULAR CRACKING OF 330 S.S. PIPING IN
H.A.Z. OF WELDED JOINTS. SUSPECTED OF BEING CORROSION FAILURES.

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I.N. | PROCESS | SEARCH CRITERIA
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344 | HYGAS | PIPING

FINAL FAILURE ANALYSIS REPORT, 12/76, BY S. DANYLUK & S. GREENBERG, ANL.
HYGAS ASH AGGLOMERATING GASIFIER INTERNAL CYCLONE DIPLEG FAILURE

SUMMARY
FAILRE: THE WALL MATERIAL OF THE AAG CYCLONE DIPLEG WAS SEVERELY
CORRODED, WHICH RESULTED IN THE DETACHMENT OF THE FLAPPER VALVE AT
THE BOTTOM OF THE DIPLEG. CARBURIZATION AND SULFIDATION OF THE DIPLEG
HAD OCCURRED WITH THE CORROSION BEING MORE SEVERE NEAR THE BOTTOM.
IN SOME AREAS 1/3 OF THE PIPE WALL WAS CONVERTED TO METAL SULFIDES.
MOLTEN SLAG WAS PRESENT NEAR THE TOP OF THE DIPLEG, CLOSE TO THE CYCLONE,
WHICH INDICATES THAT THE DIPLEG WAS EXPOSED TO OFF-DESIGN (HIGH) TEMP.
ENVIRONMENT: GASIFIER WAS OPERATED WITH BOTH STEAM AND OXYGEN AND THE DIPLEG
WAS EXPOSED TO THIS FOR ABOUT 2000 HRS, 300 HRS OF WHICH WERE IN A LOW
STEAM ATMOSPHERE. RED TEMPERATURE IS 1850-1975°F AND THE GAS COMPOSITION
30% CO2, 15% CO, 15% H2, 5% N2 AND THE BALANCE WATER.
MATERIAL OF CONSTRUCTION: INCOLOY 800
CAUSE: THE FAILURE OF THE DIPLEG IS BELIEVED TO BE ASSOCIATED WITH HIGH
TEMPERATURE (OFF-PROCESS) OPERATION WHICH LED TO SULFIDATION AND
CARBURIZATION.

RECOMMENDATIONS
1. PROCESS TEMPERATURES MUST BE KEPT WITHIN THE DESIGN LIMITS FOR INCOLOY 800.
2. USE OF A HIGHER CHROMIUM CONTENT STAINLESS STEEL SHOULD ALLEVIATE THE
SULFIDATION PROBLEM.
3. WE CONCUR WITH THE SUGGESTION BY HYGAS PERSONNEL TO ALONIZE THE FLAPPER
VALVE, WHICH MAY PROVIDE IMPROVED PERFORMANCE OF THIS COMPONENT.
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**TELECON, 12/22/76, FROM S. DANYLUK, ANL, TO B. ORCHARD, IGT.**

**STATUS OF FAILURE ANALYSES OF FAILED COMPONENTS FROM HYGAS PILOT PLANT**

### SUMMARY

1. **PROTECTION TUBE FROM AAG.**
   - Analysis has shown that the 310 S.S. thermowell was melted at the end of the tube which was inside the gasifier. Cause of failure was excessive deviation from specified temperatures.

2. **1/4" NITROGEN PURGE LINE.**
   - Embrittlement of the grain boundaries is very severe and the tube failed by brittle fracture. Sulfur content on inner phase of tube is 1-2%. Origin of sulfur unknown.

3. **ALONIZED 316 S.S. AND 446 S.S. COUPONS.**
   - Evaluation of gasifier coupons is underway.

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**FAIL. ANALYSIS RPT., 4/30/76, FROM L. ODEN, ALBANY MET. RES. CTR., TO F. GRONICKO, PERC.**

**EVALUATION OF CRACKED 316 S.S. PIPE SECTION FROM SYNTANHE PILOT PLANT**

### SUMMARY

The failed preheater pipe from the gas disengagement zone in the gas entrained feed system shows general corrosion and pitting of the inner surface in addition to severe cracking which is probably SCC. The cracking is both inter-and trans-granular.

The pipe is welded and the weld metal was attacked less severely than the parent metal. No sensitization of metal adjacent to weld was observed nor cracking of weld metal.

Electron microprobe analysis of scale shows sulfur and chlorine present in proportions of 5-10%.

316 S.S. is susceptible to chloride ions. Quantity of chlorine in scale indicates a continuous supply from the gas stream. Sulfur accentuates corrosion by decreasing the protective nature of the oxide scale. Recommend using carbon drum to remove impurities from the gas.
LETTER REPORT, 2/16/70, FROM J. LETERLE, CONOCO.
HIGH TEMPERATURE CORROSION OF PARTS FROM CO2 PILOT PLANT

SUMMARY


CONCLUSIONS

1. IT APPEARS THAT SULFUR HAS REACTED WITH ALL THREE METALS TO FORM A LOW MELTING POINT EUTECTIC OF SULFUR, CHROMIUM, NICKEL, AND IRON.
2. THE 310 S.S. HAS BEEN HIGHLY CARBURIZED.
3. IF THE ENVIRONMENT THAT CAUSED THIS CORROSION IS TYPICAL THEN CAREFUL CONSIDERATION MUST BE GIVEN TO ALLOY SELECTIONS.

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INTEROFFICE REPORT FROM H. CROWDER TO F. J. RADO, CONOCO, 8/25/70.
METALLURGICAL STUDY OF REACTOR AND GASIFIER VESSELS FROM THE LIGNITE GASIFICATION PRE-PILOT PLANT, CONSOL

SUMMARY

THE BENCH SCALE LIGNITE GASIFICATION PLANT, OPERATED AT LIBRARY, PA, HAS EXPERIENCED HIGH TEMPERATURE CORROSION AND EMBRITTLEMENT PROBLEMS. THIS REPORT SUMMARIZES WORK ON THE FOLLOWING PARTS:

1. GASIFIER VESSEL
2. REGENERATOR VESSEL
3. REGENERATOR PRE-HEAT COIL
4. REGENERATOR THERMOWELL

CONCLUSIONS: ALL FOUR OF THE PIECES HAVE EXPERIENCED ONE OR MORE OF THE FOLLOWING PROBLEMS: HIGH TEMPERATURE SULFUR CORROSION, EMBRITTLEMENT FROM CARBURIZING, EMBRITTLEMENT FROM NITRIDING.

THESE PROBLEMS COVER THE TEMPERATURE RANGE OF 950-1900°F.
FAILURE ANALYSIS REPORT, 7/25/75, BY A. MAC NAB, C. F. BRAUN CO.
THERMOCOUPLE SHEATHS-HYGAS

SUMMARY

FAILURE: THERMOCOUPLE TUBES USED IN THE STEAM-OXYGEN GASIFIER WERE BECOMING EMBRITTLE AND CORRODED AFTER SHORT EXPOSURES.

SERVICE LIFE: UNKNOWN

ENVIRONMENT: 2000F, 0.4% H2S.

MATERIAL: INCONEL 600 AND INCONEL 702.

CAUSE: "GREEN POT" CORROSION WAS THE PRIMARY MECHANISM. OCCURS IN WEAKLY OXIDIZING ENVIRONMENT. SULFIDATION ALSO OCCURRED AND THIS WAS DUE TO EXPOSURE TO REDUCING SULFIDE CONDITIONS.

RECOMMENDATIONS

1. USE AIR/NITROGEN PURGE.
2. CHANGE MATERIAL TO 310 S.S. OR INCOLOY 800.
3. LOWER NI CONTENT, INCREASE AMOUNT OF FE AND CR IN ALLOYS USED.

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I.N. PROCESS SEARCH CRITERIA
8 HYGAS THERMOCOUPLES

MEMO, 9/2/76, FROM D. DUBIS TO R. LEWIS, SYNTHANE.
THERMOCOUPLE FAILURE, GASIFIER DISTRIBUTOR CONE

SUMMARY

TELEPHONE REPORT FROM S. DANYLUK, ANL.

1. HARDNESS INDICATES COLD WORK IS PRESENT.
2. CHEMICAL ANALYSIS-INCOLOY AND MGO MEET SPECS. CORROSION PRODUCT CONTAINS 1.4 WT/%% SULFUR AND NO. 2 FUEL OIL CONTAINS 0.4 WT/%% SULFUR.
3. OPTICAL, SEM, AND MICROPROBE EXAM, CORROSION ATTACKS OD OF TUBE AND WORKS INWARD. SULFUR RICH PARTICLES FOUND IN MICROCRACKS AT FRACUTURE SURFACE.
4. INTERNAL STEAM PRESSURE GENERATED FROM WATER IN MGO OR ELSEWHERE COULD NOT CAUSE TUBE TO BURST.

RECOMMENDATIONS

1. ANNEAL TUBES BEFORE INSTALLATION (1830F/1HR).
2. AVOID FLEXING TUBES.
3. INCREASE BEND RADII WHERE TUBES ENTER PLENUM.
4. INSTALL TUBING MADE OF 310 S.S.

SEE I.N. 295, 298, 299, 300, 303, 307, 308, 312, 319 FOR ADDITIONAL DETAILS.
FAILURE ANALYSIS OF DISTRIBUTOR CONE THERMOCOUPLE SHEATHS

SUMMARY

MICROHARDNESS MEASUREMENTS INDICATE COLD WORK AT FAILURE LOCATIONS. CORROSION SCALE CONTAINS 1.4 WT % SULFUR. FABRICATOR DOES NOT UNDERSTAND SULFIDATION AT THE LOW OPERATING TEMPERATURES. LEWIS STATED THAT NO. 2 FUEL OIL CONTAINING 2.25% SULFUR WAS USED FOR START-UP AT A TEMPERATURE OF 1000 F. SINCE THIS COULD BE A PROBLEM, LEWIS SUGGESTED AND I AGREED, TO USE PROPANE AS THE START-UP FUEL.

RECOMMENDATIONS: REPLACE INCOLOY 800 WITH 310 S.S. BE CAREFUL NOT TO INTRODUCE ADDITIONAL COLD WORK IN THE MATERIAL BY BENDING THE TUBES DURING INSTALLATION.

SEE I.N. 295, 298, 299, 300, 303, 307, 308, 312, 318 FOR ADDITIONAL DETAILS.

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FINAL FAILURE ANALYSIS REPORT, 10/76, FROM S. DANYLUK & G. DRAGEL, ANL.
SYNTHANE GASIFIER DISTRIBUTOR CONE THERMOCOUPLE SHEATH (TE-268) FAILURES

SUMMARY

FAILURE: CORROSION CRACKS INITIATED AT THE OD OF THE SHEATH MATERIAL AT SURFACE CRACKS OR PITS. THE CRACKS PENETRATED TO THE ID AND THE MGO WAS EXPOSED TO STEAM. THESE CRACKS WIDENED BECAUSE OF FORMATION OF MGO(NH)2 WHICH HAS DOUBLE THE MOLECULAR VOLUME OF MGO. AS THE PROCESS CONTINUED, THE SHEATH PLASTICALLY DEFORMED AND RUPTURED ALONG THE TUBE AXIS.

ENVIRONMENT: THE THERMOCOUPLES ARE USED TO MONITOR PROCESS TEMPERATURE AND THE SHEATHS ARE NORMALLY EXPOSED TO A STEAM AND OXYGEN ENVIRONMENT, 600 PSIG AND 650F.

MATERIALS OF CONSTRUCTION: CR-AL THERMOCOUPLES WITH A SHEATH OF INCOLOY 800 AND MGO AS AN INSULATING MATERIAL.

CAUSE: A COMBINATION OF CORROSIVE ENVIRONMENT, OFF-PROCESS TEMPERATURES, SHEATH FABRICATION AND POSSIBLE COLD WORKING OF THE SHEATHS CONTRIBUTED TO THE FAILURES.

RECOMMENDATIONS

1. BETTER QUALITY CONTROL OF SHEATH FABRICATION. USE STANDARD (SPEC. C7-6T) FOR METAL SHEATH, CERAMIC INSULATED THERMOCOUPLES.
2. A HIGHER CR S.S. (TYPE 310) SHOULD SOLVE THE SULFIDATION PROBLEM.
3. SINCE THE FUEL OIL FOR START-UP CONTAINS SULFUR, SWITCH TO PROPANE.
4. CAREFUL HANDLING OF TUBES AND ANNEALING PRIOR TO INSTALLATION WOULD ELIMINATE THE COLD WORK.

SEE I.N. 295, 298, 299, 300, 303, 307, 308, 312, 318, 319, FOR PREVIOUS REPORTS.
I.N. PROCESS SEARCH CRITERIA
337 SYNTHANE THERMOCOPLES

LETTER W/COPY OF TC STANDARDS, 11/4/74, FROM S. DANYLUK, ANL, TO B. ROCHE, LUMMUS.
ROD STANDARD C7-6T W/SUPPLEMENTS

SUMMARY

THERMOCOUPLE MATERIAL AND THERMOCOUPLE ASSEMBLY
CHROMEL-P VERSUS ALUMEL
STAINLESS STEEL SHEATHED
MAGNESIUM OXIDE INSULATED
SEE SECTION ON SURFACE FINISH AND DEFECTS. IT DOES NOT APPEAR THAT THE
THERMOCOUPLE SHEATHS WE HAVE EXAMINED WOULD HAVE MET THESE SPECIFICATIONS.
IT MIGHT BE A GOOD IDEA TO USE THESE Specs AS A GUIDE WHEN ORDERING FUTURE
 MATERIAL.

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I.N. PROCESS SEARCH CRITERIA
341 SYNTHANE THERMOCOPLES

TELECON, 11/29/76, FROM S. DANYLUK, ANL, TO B. MASSA, LUMMUS.
SUBSTITUTE MATERIALS FOR 310 S.S. TO BE USED IN GASIFIER

SUMMARY

310 S.S. WAS RECOMMENDED AS A SUBSTITUTE FOR INCOLOY 900 THERMOCOUPLE
SHEATHS WHICH WERE FAILING IN SERVICE. HOWEVER, 310 S.S. SHEATHS WILL NOT
BE AVAILABLE FOR SEVERAL MONTHS. 446 S.S. WAS RECOMMENDED BUT EMBRITTLEMENT
MAY BE A PROBLEM.
OTHER
PROJECT ADVISORS MEETING, 5/21/74, NO AUTHOR.
DISCUSSION ON PROBLEMS AT CO2 PILOT PLANT.

SUMMARY

BALL VALVES: RAPID CITY USES CARBON SEATS WITH S.S. BALLS.
RAW COAL STORAGE BIN: FREEZE UP WILL OCCUR. RECOMMEND STEAM TRACING OR ELECTRICAL TRACING.
INERT GAS COMPRESSOR AFTERCOOLER: CORROSION PROBLEMS DUE TO NITRIC OXIDES FORMING UNDER PRESSURE AND IN THE PRESENCE OF WATER. ORIGINAL MATERIAL, ADMIRALTY, NEW MATERIAL, 316 S.S. ELIMINATION OF MOISTURE APPEARS TO BE THE SOLUTION.
GASIFIER OUTER SHELL: TEMP. SENSITIVE PAINT (400-500 F RANGE) SHOULD BE APPLIED TO OUTER SHELL TO DETECT HCT SPOTS.
PRETREATER: POTENTIAL SCC COULD OCCUR IN PRETREATER DUE TO CHLORIDES FROM THE FEED. 316 S.S. MIGHT CURE THE PROBLEM.
RAYMOND CYCLONE SEPARATOR: ABRASION RESISTANT LINING SHOULD BE APPLIED TO THE INTERNAL SURFACE.

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TELECON, 6/3/76, FROM S. GREENBERG, ANL, TO B. BAIR, IG.
ASH AGGLOMERATING GASIFIER, INTERNAL CYCLONE DIP LEG

SUMMARY

CORROSION WAS MORE SEVERE AT BOTTOM. A SAMPLE WAS REMOVED FROM TOP SECTION FOR MAT' L IDENT. AND METALLOGRAPHIC EXAM. VISUAL EXAM. OF THIS SAMPLE REVEALED AREAS OF DEEP PENETRATION OF THE INSIDE WALL BY CORROSION. ONE AREA HAD "1/3 OF W.T. PENETRATED BY CORROSION. EXAM. AT 30X MAG. CLEARLY SHOWED THAT MOLTEN MATERIAL HAD BEEN IN CONTACT WITH WALL SURFACE. PRESENCE MOLTEN SLAG IS PROBABLY INDICATIVE OF OFF-DESIGN PERFORMANCE.
IN PROCESS SEARCH CRITERIA

297 SYNTHANE AUXILIARY PROCESS EQUIPMENT

ENGINEERING INSPECTION REPORT, 7/19/76, BY J. JEWELL, LUMMUS.
RAW GAS SCRUBBER.

SUMMARY

HOLD DOWN TRAY: CONSIDERABLE CORROSION, RESULTING IN MANY HOLES. HOLES ARE LARGE ENOUGH FOR PALLS TO PASS THROUGH, THUS THE TRAY CANNOT PERFORM ITS TASK.

PACKING PALLS: POOR CONDITION AND CORRODED EXTENSIVELY. PARTS ARE MADE FROM CARBON STEEL AND ARE NOT COMPATIBLE WITH GASSES AND LIQUIDS, THUS SEVERE CORROSION OCCURS. PLANS ARE TO REPACK THE VESSEL WITH 316 S.S. PALLS AND INSTALL NEW RetAINING PLATES, IF NEEDED, ALSO OF 316 S.S.
SEE I.N. 309 FOR FURTHER INFORMATION.

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I.N. PROCESS SEARCH CRITERIA

309 SYNTHANE AUXILIARY PROCESS EQUIPMENT

REPORT, 8/12/76, FROM J. JEWELL, LUMMUS.
DA-201 RAW GAS SCRUBBER VESSEL WALL CORROSION.

SUMMARY

INSPECTORS OBSERVED SEVERE CORROSION OF PALL RINGS AND HOLD DOWN PLATES IN RAW GAS SCRUBBER. THIS REPORT GIVES RESULTS OF ULTRASONIC THICKNESS MEASUREMENTS. THESE SHOW A LOSS OF THICKNESS UP TO 0.035-IN FROM AN ORIGINAL 1-1/8 IN MINIMUM WALL THICKNESS (43). DATA READINGS ARE ATTACHED. FUTURE READINGS WILL BE MADE TO PLOT PROGRESS OF CORROSION TO HELP IN ASCERTAINING VESSEL LIFE.
SEE I.N. 297 FOR FURTHER INFORMATION.
FAILURE: THE FLOW IN THREE PASSES OF THE FURNACE WAS CONSTRUCTED DUE TO VERY SEVERE SCALING. THE RUN TERMINATED DUE TO EXCESSIVE PRESSURE DROP ACROSS THE COIL DUE TO ABOVE SCALING.

SERVICE LIFE: 1699 HRS
ENVIRONMENT: TEMP. 1100-1400°F. GAS COMPOSITION-3-4% CO, 26% CO2, 70% N2, CARBONYL SULFIDE 50 PPM NOMINAL (RANGES FROM TRACE TO 400 PPM).

MATERIAL OF CONSTRUCTION: INCOLOY 800

CAUSE OF FAILURE: UNKNOWN

ACTION: DIAGNOSTIC ANALYSIS TO BE PERFORMED BY CONOCO.*

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I.N. PROCESS SEARCH CRITERIA
130 CO2 PIPING

PAPER, 3/19/73, BY K. YOUNGSLUT, IGT. NACE CONFERENCE, CORROSION/73, ANAHEIM, CA.
EXPERIENCE WITH MATERIALS IN HYGAS PILOT PLANT

SUMMARY

DETILED GENERAL REVIEW PAPER ON HYGAS MATERIAL PROBLEMS.

1. LOW PRESSURE OIL/COAL SLURRY PUMP: CENTRIFUGAL TYPE. CAST STEEL WITH PLASMA SPRAYED COATING. COATING ERODED AWAY THEN STEEL PARTS ERODED. PARTS WERE THEN OVERLAI WITH STELLITE 12 AND THE PUMP SPEED REDUCED BY 1/2. PROBLEM HAS BEEN MINIMIZED.

2. HIGH PRESSURE OIL/COAL SLURRY PUMP: RECIPROCATING "MUD PUMPS" WERE SELECTED. CHECK VALVE SEALS ARE A PROBLEM. USING VITON (BEST MATERIAL FOUND) VALVE LIFE RANGES FROM A FEW HOURS TO A FEW DAYS. NOW USING VALVES COATED WITH STELLITE AND SEAT RINGS OF STELLITE. VALVE DESIGN STILL NEEDS TO BE IMPROVED.

3. HYDROGASIFIER INTERALS: 1500 PSI, 600-1800 F, H2 TO 650 PSI. FAILURES IN 446 S.S. PIPING REPLACED WITH INCOLOY 800. WHEN USING HIGH SULFUR COALS THE INCOLOY 800 WILL BE REEVALUATED.

4. PRODUCT GAS QUENCH SYSTEM: ALL WETTED PARTS OF CENTRIFUGAL PUMPS OVERLAI WITH 304 S.S. ANHYDROUS AMMONIA ADDED FOR PH CONTROL. SUCCESSFUL CHANGE.

5. HIGH PRESSURE WATER/CHAR SLURRY PUMP: THE USE OF STELLITE 12 OVERLAY AND PUMP SPEED REDUCTION HAS BEEN SUCCESSFUL IN MINIMIZING PROBLEMS.
MEMO, 4/29/76, FROM S. GREENBERG TO R. WEEKS, ARGONNE.
FAILED HYGAS COAL PRETREATMENT VESSEL COOLER (NO. 2)

**SUMMARY**

SECOND FAILED COOLER. FAILURE IN 1ST COOLER WAS MECHANICAL WHILE THIS NEW FAILURE IS SEVERELY CORRODED. FAILURE APPEARS TO BE THE RESULT OF RUPTURE OF A CORROSION-THINNED PIPE WALL. A DETAILED OPERATING HISTORY IS EXPECTED FROM HYGAS ALONG WITH A CLINKER SAMPLE. A DETAILED METALLOGRAPHIC EXAM. OF FAILED COOLER WILL BE PERFORMED.

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ERDA MAT. AND COMP. FAIL. REPORT, 5/7/76, FROM W. ORCHARD, IGT.
COOLING BUNDLE FROM PRETREATER REACTOR

**SUMMARY**

FAILURE: COOLING BUNDLE REMOVED AFTER TEST NO. 50. TWO TUBES HAD SMALL RUPTURES. SEVERAL TUBES WERE BENT. LARGE AREAS OF YELLOWISH-ORANGE DEPOSITS ON OUTER SURFACE OF COOLING BUNDLE.
SERVICE LIFE: 1350 HRS AT TEMP. 30 THERMAL CYCLES.
MATERIAL: SA-106-GR B CARBON STEEL
ENVIRONMENT: 300 F FLUIDIZED BED (AIR AND BITUMINOUS COAL, STEAM). SUPERFICIAL VELOCITY, 1/2 FT/SEC. BED DENSITY, 25 LB/CU. FT. PARTICLE SIZE RANGE, -8 TO +200 MESH.
CAUSE: ONE OF THE HOLES SEEMS TO HAVE BEEN CAUSED BY A STEAM JET FROM THE OTHER HOLE. THE FIRST HOLE LOOKS TO HAVE RUPTURED DUE TO WALL THINNING.
ACTION: ANALYSIS BY ANL.
ERDA MAT. AND COMP. FAIL. REPORT, 5/7/76, FROM W. ORCHARD, IGT.
THERMOWELLS AND PRESSURE TAPS

SUMMARY

FAILURE: THERMOWELLS AND PRESSURE TAPS WERE FOUND TO BREAK OFF IN SERVICE
AFTER EXPERIENCING SEVERE CORROSION AND EVIDENCE OF LOCAL MELTING.
MATERIAL: RA 330 PIPE, VARIOUS DIAMETERS.
SERVICE LIFE: SEE ATTACHMENTS.
ENVIRONMENT: SEE ATTACHMENTS.
POSSIBLE CAUSE: PROBLEM COULD BE RELATED TO A NEW FEEDSTOCK MATERIAL
WHICH CONTAINS MORE SULFUR THAN THE PREVIOUS FEED.
ACTION: FAILURE ANALYSIS TO BE PERFORMED BY ANL.

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I.N. PROCESS SEARCH CRITERIA
209 HYGAS PIPING

TELECON, 5/18/76, FROM S. GREENBERG, ANL, TO B. ORCHARD, IGT.
GASIFIER TRANSFER LINE BELLOWs AND AAG DIPLeg.

SUMMARY

ANOTHER EXPANSION JOINT FAILED AND WILL BE SENT FOR ANALYSIS WHEN RETURNED
FROM MANUFACTURER.
INITIAL EXAM. OF CRACKED PIPE THOUGHT TO BE INCOLOY 800 (ACTUALLY 330 S-S.)
INDICATED THAT CRACKING WAS INTERGRANULAR AND THAT CORROSION WAS CONSIDERED
TO BE AT LEAST A CONTRIBUTING FACTOR.
AN INTERNAL CYCLONE DIPLeg FROM THE ASH AGGLOMERATING GASIFIER FAILED
AND WILL BE SENT FOR FAILURE ANALYSIS.
FAILURE: TUBING IS BRITTLE AND WILL NOT BEND.
SERVICE LIFE: UNKNOWN
ENVIRONMENT: STEAM-OXYGEN ZONE OF HYGAS REACTOR. ABOUT 1600 F. INSIDE OF TUBE USUALLY PURGED CONTINUOUSLY WITH NITROGEN.
MATERIAL: 316 S.S. 1/4" O.D. X 0.049" WALL THICKNESS. G
CAUSE: VISUAL INSPECTION SUGGESTS GRAIN GROWTH AND CORROSION OF THE INSIDE SURFACE OF THE TUBE.
ACTION: REPLACED PART. ANALYSIS BY ARGONNE NATIONAL LAB.

SUMMARY REPORT OF METAL FAILURES WHICH HAVE OCCURRED AT PROJECT LIGNITE FROM START-UP TO PRESENT (7/24/75).
OPERATING CONDITIONS FOR THE THREE PREHEATER COIL FAILURES ARE GIVEN.
STATUS REPORT OF FAILURE SAMPLE DEPOSITION AND STATUS OF FAILURE ANALYSES BOTH FROM INPLANT AND OUTSIDE CONSULTANTS (RALPH PARSONS & GULF R & D).
RECOMMENDATIONS ARE MADE FOR IMPROVING FAILURE REPORTING AND FAILURE ANALYSIS.
ABSTRACT: FIRESIDE CORROSION IN MUNICIPAL INCINERATORS VERSUS REFUSE COMPOSITION

SUMMARY

CORROSION OF HEAT RECOVERY TUBES EXPOSED TO COMBUSTION PRODUCTS FROM THE BURNING OF MUNICIPAL AND INDUSTRIAL WASTES HAS BEEN A MAJOR PROBLEM. THE RESEARCH PROGRAM REPORTED HERE SOUGHT A BETTER UNDERSTANDING OF THE FACTORS THAT CONTRIBUTE TO THE CORROSION OF STRUCTURAL AND ENERGY-RECOVERY SYSTEMS ASSOCIATED WITH MUNICIPAL REFUSE INCINERATORS. THE RESULTS SHOW THAT THE SULFUR AND CHLORINE CONTENTS OF REFUSE HAVE SIGNIFICANT EFFECTS ON THIS TYPE OF CORROSION ACCELERATING WHEN THE CHLORIDE CONTENT OF THE REFUSE INCREASED, BUT SLOWING DOWN WHEN THE SULFUR WAS INCREASED.

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LETTER REPORT, 10/27/76, FROM R. COOPER, ORNL, TO D. BIENSTOCK, PERC.
ANALYSIS OF NINE CORROSION SAMPLES

SUMMARY

MATERIAL CHARACTERIZATION: CHEMICAL ANALYSIS OF THE BASE METAL AGREED WITH VENDORS CERTIFICATION.
STEAM SIDE CORROSION: CARBON STEELS IN STEAM ENVIRONMENTS AT MODERATE TEMP. USUALLY FORM A PROTECTIVE SCALE OF MAGNETITE. THE LOCALIZED PITTING AND RAPID SCALE GROWTH ON THESE SAMPLES IS BELIEVED TO BE THE RESULT OF ATMOSPHERIC EXPOSURE ASSOCIATED WITH BOILER SHUTDOWN AND SUBSEQUENT HANDLING OF THE TUBES FOLLOWING DISASSEMBLY. THIS IS A SERIOUS PROBLEM IN COMMERCIAL BOILERS AND IS A COMMON CAUSE OF PREMATURE FAILURE OF THE BOILER TUBES.
FLUE GAS CORROSION: BOILER COMBUSTION ZONE WAS OPERATED WITH EXCESS AIR VARYING BETWEEN 20-35%. FLUE GAS ANALYSIS SHOWED SIGNIFICANT LEVELS OF CO2, H2O AND TRACES OF SO2. SCALE FORMED IN THIS ENVIRONMENT IS NOT AS TENACIOUS AND PROTECTIVE AS SCALE FORMED IN STEAM. THEREFORE THERE WILL BE SIGNIFICANTLY HIGHER CORROSION RATES. RUST ON INSIDE SURFACES AVERAGED 3 MILS IN THICKNESS (PROJECTED CORROSION RATE OF 12.5 MILS/YEAR).
FINAL REPORT WILL FOLLOW.
### Operational and Mechanical History Summary

1. **Stuffing Box/Check Valve Failures**
2. **Initial Loss of Metal Due to Erosion/Corrosion in Slurry Preheater Coil** — Then metal loss arrested.
3. **Erosion of Control Valves**
4. **Mechanical Problems in Filtration**
5. **Severe Erosion and Seal Leakage in All Screw Pumps Handling Diatomaceous Earth** — Replace with Piston or Centrifugal Pumps
6. **Many Seal Failures on Pumps**
7. **Plugging of Piping Systems** — Heat to 500°F to solve.
8. **Corrosion of Solvent Recovery System** — Replace carbon steel with S.S.

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### Corrosion Test Results

Corrosion racks were installed in wash solvent column to evaluate materials. Type 321 S.S. and Incoloy 800 showed very good resistance to the corrosive environment. Moly bearing alloys (2-3%) are thought to have good corrosion resistance to napthenic acid corrosion. See I.N. 302 for further information.
FAILURE: COLUMN SHELL DEVELOPED A LEAK DUE TO CORROSION.

SERVICE LIFE: 18 MONTHS.

PRIOR REPAIRS: REBOILER RETUBED, 4/75 NEW TRAYS INSTALLED 12/75.

ENVIRONMENT: OVERHEAD, 450-525°F, WASH SOLVENT. BOTTOMS, 600-670°F, PROCESS SOLVENT, 10PSIG.

MATERIAL: SA-515 GR. 55 CARBON STEEL.

CAUSE: CORROSIVE ATTACK SIMILAR TO THAT OF NAPHTHENIC ACID CORROSION.

ACTION: REPLACED TOP 18-FT SECTION WITH 1/4-IN 316L S.S.

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FAILURE REPORT, 7/28/76, FROM D. CANFIELD TO L. SAMUELS, FT. LEWIS.

FAILURE OF THE WASH SOLVENT COLUMN

SUMMARY

THE WASH SOLVENT COLUMN DEVELOPED A LEAK BETWEEN THE 11TH AND 12TH TRAYS.

CAUSE AND DAMAGES THE SAME AS PREVIOUS FAILURE (SEE FAILURE REPORT IN I.N. 267).

THE LEAK WAS FIXED WITH A TEMPORARY PATCH. A MAJOR SHUTDOWN IS SCHEDULED FOR NOVEMBER AND THE REST OF THE CARBON STEEL COLUMN WILL BE REPLACED AT THAT TIME WITH 316L S.S.

AN INSPECTION OF THE 316L S.S. SHELL AND TRAY SUPPORTS INSTALLED 3/76 REVEALED NO NOTICEABLE CORROSION. THE 316 S.S. VALVES INSTALLED AT THE SAME TIME ALSO APPEARED TO BE IN EXCELLENT SHAPE.

SEE I.N. 267 AND 314 FOR ADDITIONAL DETAILS.
I.N. PROCESS SEARCH CRITERIA
314 SRC PIPING

ERDA MAT. AND COMP. FAIL. REPORT, 7/29/76, FROM J. PIATT, FT. LEWIS.
WASH SOLVENT COLUMN 04075302

SUMMARY

FAILURE: COLUMN SHELL DEVELOPED A LEAK.
SERVICE LIFE: 22 MONTHS
ENVIRONMENT: OVERHEAD, 450-525F, WASH SOLVENT. BOTTOMS, 600-670F, PROCESS
SOLVENT, 10 PSIG.
PRIOR REPAIRS: REBOILER RETUBED 4/75. NEW TRAYS INSTALLED 12/75.
REPLACED TOP 18' WITH 316L S.S., 4/1/76
MATERIAL: SA-515 Gr. B CARBON STEEL
CAUSE: CORROSIVE ATTACK SIMILAR TO NAPHTHENIC ACID CORROSION.
ACTION: PATCHED HOLE. WILL REPLACE REMAINING CARBON STEEL COLUMN WITH 1/4" 
316L S.S. DURING A MAJOR SHUTDOWN LATE THIS YEAR.
SEE I.N. 267, 315 FOR FURTHER INFORMATION.

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I.N. PROCESS SEARCH CRITERIA
208 HYGAS PIPING

TELECON, 5/24/76, FROM S. GREENBERG, ANL, TO B. BAIR, IGT.
ASH AGGLOMERATING GASIFIER-INTERNAL CYCLONE DIPLEG

SUMMARY

INITIAL INSPECTION OF THE DIPLEG REVEALED VERY SEVERE CORROSION, PARTICULARLY 
at the bottom. 310 S.S. IS RECOMMENDED FOR THE REPLACEMENT DIPLEG WITH 
THE FLAPPER VALVE ALONIZED.
THE PROBLEM WITH FAILURES LIKE THE DIPLEG AND EXPANSION RELLOWS IS THAT 
HYGAS IS OPERATING BEYOND THE EDGE OF EXISTING TECHNOLOGY AND THE NEED IS 
FOR NEW MATERIALS DEVELOPMENT NOT MERELY TROUBLESHOOTING.
### Purge Piping Corrosion—Gasifier

**Summary**

**Failure:** Corrosion observed on purge piping located in gasifier BR-1 or BR-3. 
**Service Life:** 114 hrs. 
**Action:** Analysis to be performed by Argonne.

### Waste Water Line—1” Schedule 40

**Summary**

**Failure:** Pipe failed at root of thread due to erosion, corrosion and vibration. 
**Service Life:** 18 months. 
**Environment:** Internal, coal tar liquor at 180 F. 
**Material:** Carbon steel. 
**Recommendation:** Future connection to be socket welded rather than threaded. 316 S.S. Schedule 90 pipe will be used. 
**Action:** Part replaced. No analysis.
FAILURE: PIPE FAILED AT ROOT OF THREAD DUE TO CORROSION AND VIBRATION.
SERVICE LIFE: 13 MONTHS
ENVIRONMENT: CHLORINATED COAL TAR LIQUOR
MATERIAL: CARBON STEEL
RECOMMENDATION: ELIMINATE THREADS, USE S.S.
ACTION: REPLACED PART. ANALYSIS TO BE PERFORMED BY COMPANY LAB.
A REVIEW STUDY OF FIRESIDE CORROSION IN UTILITY AND INDUSTRIAL BOILERS.

METHODS TESTED AND RECOMMENDED FOR THE PRACTICAL PREVENTION OF CORROSION INCLUDE FURNACE OPERATIONAL CHANGES, FUEL TREATMENT, ADDITIVES, PROTECTIVE COATINGS, ALLOY SELECTION, AND DESIGN CHANGES.

CORROSION OF FOSSIL FUELED STEAM GENERATORS.

PAPER DISCUSSES THE PROBLEM OF OPERATIONAL CORROSION IN HIGH PRESSURE, DRUM-TYPE STEAM GENERATORS. PAST EXPERIENCE IS REVIEWED. PRESENTED IS A THEORY OF BOILER CORROSION BASED ON THE ASSUMPTION THAT SPECIFIC MECHANICAL AND CHEMICAL CONDITIONS MUST EXIST SIMULTANEOUSLY TO INITIATE CORROSIVE ATTACK. EMPHASIS IS PLACED ON UNDERSTANDING THE FLUID DYNAMICS AND HEAT TRANSFER CONDITIONS WHICH MAY EXIST WITHIN A BOILER TUBE IN DIFFERENT LOCATIONS IN THE BOILER. RECOMMENDATIONS FOR CHEMICAL CONTROL AND BOILER DESIGN TO MINIMIZE THE PROBLEM OF BOILER CORROSION ARE INCLUDED.
ERDA MAT. AND COMP. FAIL. REPORT, 8/6/76, FROM J. JEWELL, LUMMUS.
GASIFIER THERMOCOUPLE TE 268

SUMMARY

FAILURE: SHEATH SEPARATED, OPENED UP, EXTREME CORROSION.
SERVICE LIFE: 17 HRS
ENVIRONMENT: 600 PSI, 500F, STEAM AND OXYGEN-SOME ASH.
MATERIAL: INCOLOY 800
CAUSE: SHEATH MATERIAL VERY REACTIVE TO ENVIRONMENT.
ACTION: REPLACED. DIAGNOSIS TO BE PERFORMED BY ANL.

I.N. PROCESS SEARCH CRITERIA
308 SYNTHANE THERMOCOUPLES

ERDA MAT. AND COMP. FAIL. REPORT, 10/19/76, FROM J. JEWELL, BRUCETON.
GASIFIER CONE THERMOCOUPLE TE-268

SUMMARY

FAILURE: SHEATH CORRODED AWAY
PRIOR REPAIRS: THIRD THERMOCOUPLE ASSEMBLY TO FAIL
MATERIAL: SHEATH MADE OF 304 S.S.
ACTION: REPLACED WITH 310 S.S. SHEATH. ANALYSIS BY COMPANY LABS.
I.N. PROCESS SEARCH CRITERIA

305 WESTINGHOUSE THERMOCOUPLES

ERDA MAT. AND COMP. FAIL REPORT, 7/26/76, FROM E. VANDERGRIFT, WESTINGHOUSE.
THERMOCOUPLE TE 502-1

SUMMARY

FAILURE: SCRATCH IN SECONDARY COATING LED TO CORROSION OF SECONDARY SHEATH AND BREAKDOWN OF INSULATION RESULTING IN ERRATIC OUTPUT.

SERVICE LIFE: 104 HRS

ENVIRONMENT: 2000°F OXIDIZING AND REDUCING GASES

MATERIALS: SIC PRIMARY SHEATH, DISILICIDE COATED MD SECONDARY SHEATH, MgO INSULATION, W5RE/W26RE THERMOCOUPLE.

CAUSE: MANUFACTURING PROCESS CAUSED CRIMP IN SECONDARY SHEATH WHICH LED TO ULTIMATE FAILURE BY PENETRATION OF TOXIC GASES.

ACTION: ANALYSIS BY COMPANY LABS
A REVIEW OF LETDOWN VALVE EXPERIENCE IN COAL LIQUEFACTION SERVICE

SUMMARY

1. WEAR OF THE HIGH PRESSURE LETDOWN VALVE.

2. LITERATURE SURVEY COVERING LIQUEFACTION PROCESSING TECHNOLOGY. THIS INCLUDES THE TECHNOLOGY OF THE CORROSIVE-EROSIVE WEAR PROCESS AND OF PRESENT LETDOWN VALVE TRIM.

3. HISTORY OF EARLY EXPERIMENTAL COAL LIQUEFACTION PROCESSES FROM GERMANY AND UNITED STATES.

4. LITERATURE REVIEW OF EROSIVE WEAR ON DUCTILE, BRITTLE AND COMPOSITE CEMENTED CARBIDE MATERIALS AND THE PROPOSED OPERATIVE MECHANISMS.

5. THE CEMENTED CARBIDES USED IN TRIM COMPONENTS SEEM TO HAVE A MARKED DIFFERENCE IN QUALITY AND COMPOSITION FROM SUPPOSEDLY IDENTICAL GRADINGS OF CARBIDE.

6. DISCUSSES THE VARIOUS DESIGNS OF TRIM IN USE, METHOD OF THROTTLING, PROCEDURES USED TO MODIFY VALVES, AND OTHER WEAR PROBLEMS IN LIQUEFACTION SYSTEMS.

* * * *
This report consists of a group of summaries of operating experiences at coal conversion pilot plants, materials evaluation reports, and diagnostic failure analysis reports dealing with the problem of corrosion in the coal conversion industry.

**KEY WORDS** (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)

Coal gasification; coal liquefaction; component failures; corrosion; failure analysis; material evaluation

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