Report of Operating Experiences in Coal Conversion Plants Material of Construction - Incoloy 800

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.
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 database 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.

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(1) The Newsletter is published by Battelle, Columbus Labs, 505 King Avenue, Columbus, Ohio 43201. Editor: R. Schorr.
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<td>BCR BI-GAS Pilot Plant</td>
<td>Converts coal to high BTU gas</td>
<td>Stearns-Roger, Inc. Homer City, PA</td>
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<td>BMI</td>
<td>Battelle, Columbus The Agglomerating Burner Gasification PDU</td>
<td>Converts coal to synthetic gas</td>
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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 coal to low sulfur, low ash coke and liquid and gaseous fuels</td>
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<td>COED</td>
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<td>Converts coal to low sulfur crude oil and medium BTU gas</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>
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<td>Converts high sulfur, high ash content coal to an ashless, low-sulfur solid fuel</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 use of Incoloy 800 in coal conversion processes. The Incoloy 800 summaries are categorized into subgroups of specific components: auxiliary process equipment, piping, pressure vessels, thermocouples, valves and material evaluation. Each component subgroup is sorted by coal conversion process (such as CO2, Hygas, 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.
AUXILIARY

PROCESS

EQUIPMENT
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 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.

* * * * *
I.N. PROCESS SEARCH CRITERIA
311 SYNTHANE AUXILIARY PROCESS EQUIPMENT

ERDA MAT. AND COMP. FAIL. REPORT, 3/12/76, FROM J. JEWELL, LUMMUS.
INTERMEDIATE HEAD 0C-202

SUMMARY

FAILURE: HEAD BUCKLED INTO REVERSE DISH.
SERVICE LIFE: UNKNOWN
ENVIRONMENT: AMBIENT, CO2 GAS, OPEN TO AIR. MAX. PRESS. 80 PSIG.
CAUSE: ALLOWABLE DIFFERENTIAL PRESSURE WAS EXCEEDED.
ACTION: FAILURE ANALYSIS TO BE PERFORMED BY LUMMUS CO.
I.N. PROCESS SEARCH CRITERIA
378 SYNTHOIL AUXILIARY PROCESS EQUIPMENT

LETTER REPORT, 4/27/77, FROM T. COX TO J. BATCHELOR, ERDA.
REVIEW OF MATERIALS OF CONSTRUCTION FOR SYNTHOIL PDU

SUMMARY

EXERTS FROM ORNL, ANL, NBS, AND THE MATERIALS BRANCH OF MER HAVE EXAMINED CERTAIN CRITICAL AREAS OF THE SYNTHOIL PDU. GENERAL CONSENSUS IS THAT THE MATERIAL SELECTION IS QUITE GOOD. SOME TROUBLE SPOTS ARE PINPOINTED.

POTENTIAL PROBLEM AREAS

1. FROM THE EXPERIENCE AT THE SRC PILOT PLANT, CASINGS AND IMPELLERS IN THE CENTRIFUGAL PUMPS WILL RAPIDLY DETERIORATE. DESIGN OF THESE PUMPS SHOULD BE REVIEWED.

2. HIGH PRESSURE HYDROGEN LINES: UNTIL THE SAFETY OF CARBON STEEL TUBING CAN BE ESTABLISHED BY APPROPRIATE TESTS, THESE LINES SHOULD BE CONSTRUCTED OF 304L S.S.

3. "HAIRPIN" HEAT EXCHANGERS: POORLY DESIGNED FROM AN EROSION AND FABRICATION VIEWPOINT.

4. REACTOR FEED HEATER PIPING: INLET PIPING DESIGN COULD LEAD TO EROSION FROM TURBULENT FLOW INDUCED BY HYDROGEN.

5. MESH SCREENS: CONTAIN CATALYSTS IN REACTOR FEED SURGE VESSEL AND THE REACTORS. THEY SEEM TO BE VULNERABLE TO EROOSIVE WEAR. DESIGN SHOULD BE CHANGED TO ALLOW EASY REPLACEMENT.

* * *
SCIENTIFIC PAPER, 11/25/75, BY L. ALBERTIN AND J. CUNNINGHAM, WESTINGHOUSE AND S. TENDULKAR AND P. CHERISH, ENERGY SYSTEMS.
MATERIAL PROBLEMS IN A SYNTHESIS GAS GENERATOR FOR A MULTI-STAGE COAL GASIFICATION SYSTEM

SUMMARY
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.

* * * * *
MATERIAL EVALUATION
CORROSION TEST SPOOL-6376

SUMMARY

TWELVE ALLOYS WERE TESTED FOR CORROSION RESISTANCE UNDER THE FOLLOWING CONDITIONS:
PROCESS UNIT: 3 GALLON RETORT AT 120 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 120 PSIG
CORROSIVE MEDIA: COAL GASIFICATION PROCESS QUENCH TANK LIQUOR
AVERAGE TEMPERATURE: 350°F
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.
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, 1900°F MOLTEN SODIUM CARBONATE (FROM THE OCR COAL GASIFICATION PROGRAM) FOR 100 HRS. THE CERAMICS AL2O3, ZR02, MGO, AND BEO AND THE METALS PLATINUM AND UNILOY 50491 WERE FOUND TO BE THE MOST CORROSION RESISTANT MATERIALS.

REPORT, 10/1/75, BY P. 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 PSI. 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.
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 1000F INCLUDING 24 HRS OVER 1300F. EXAM. SHOWED SIGNIFICANT CARBURIZATION (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 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 DEFENTRAIMENT 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 3A 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 3B. 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.
I.N.  PROCESS SEARCH CRITERIA
169  CLEAN COKE MATERIAL EVALUATION

LETTER, 6/28/72, FROM E. PHELPS, CORROSION TECHNOLOGY, TO P. MASCIAntonio, USS CORP
MATERIALS OF CONSTRUCTION FOR THE CLEAN COKE PROJECT CARBONIZATION PDU

SUMMARY

AFTER REVIEWING FLOW DIAGRAMS AND PROCESS CONDITIONS FOR THE SUBJECT EQUIPMENT
THE FOLLOWING MATERIALS OF CONSTRUCTION ARE RECOMMENDED.
304 S.S., 316 S.S., CARBON STEEL, INCOLOY 800.
SEE TABLES I-IV FOR SPECIFIC RECOMMENDATIONS.

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I.N.  PROCESS SEARCH CRITERIA
183  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.
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 108% 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 (9/75).

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. BODY, 410 S.S. GLAND NUT, 416 S.S. COLLAR). DESIGN CHANGED TO MAKE ALL PARTS FROM 316 S.S.
I.N. PROCESS SEARCH CRITERIA
180 COED MATERIAL EVALUATION

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 600-700°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 700-800°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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14
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 CALCINEP AT LIBRARY, PA.
ATMOSPHERE: SULFUR BEARING GAS AT HIGH TEMPERATURES.
APPLICATIONS
GASIFIER-DEVOLATILIZER INTERNALS: CYCLONES, DIPEGS, THERMOWELLS, PRESSURE TAP PROBES, HEATERS. TEMPERATURE RANGE 1500-1600F. SULFUR AS H2S, 0.1%.
310 S.S. AND INCOLOY 800 CAN BOTH BE USED.
REGENERATOR INTERNALS: TEMP/PRESS PROBES, THERMOWELLS. TEMPERATURE RANGE 1900-1950F. SULFUR ABOUT 0.2% AS SO2, H2S, S2, COS. NO ALLLOY SUITABLE.
EXTERNAL PROCESS PIPING: HIGH TEMPERATURE HEATERS, SOLIDS TRANSFER LINES. TEMPERATURE RANGE 1500-1950F. SULFUR FROM 0-0.1% AS H2S. USE 310 S.S. OR INCOLOY 800 EQUALLY.

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.
SUMMARY

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

CONCLUSIONS

1. THE ALONIZED LAYER ON INCOLOY 300 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 1330°F 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 1650°F, A RADIUS OF 6 IN AT 1300°F, OR A RADIUS OF 26 IN AT 1000°F.

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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SUMMARY

ALONIZED INCOLOY 800 PREHEATER COIL FABRICATION GUIDELINES

SUMMARY OF CONOCO RESEARCH REPORT 72-74-1301 OF THE STUDY MADE TO YIELD GUIDELINES FOR BENDING ALONIZED TUBING FOR PREHEATER COILS.
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.

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REPORT, N.D., BY J. DEVAN, P. 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 ASSESS 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 UNILLOY 50/50) OR ALONIZED 310 S.S. OR
ALONIZED INCOLOY 800, 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 671/800H 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.
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.
FAILURE REPORT: THE SHELL OF THE 3/8" CARBON STEEL (SA-515 GR. 55) COLUMN STARTED TO LEAK BETWEEN THE 18TH 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 DOWNCOMER SUPPORTS FROM TRAYS 12-21 WERE ALMOST COMPLETELY CORRODED. 2) AREAS OF LIQUID PHASE CONTACT WERE CORRODED MORE THAN THOSE IN CONTACT WITH VAPOR PHASE, 3) 14 GAUGE 321 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 NAPHTHENIC ACID CORROSION AS DESCRIBED IN METALS HANDBOOK, VOL. 1. HOWEVER, NO DEFINITE CONCLUSIONS ARE MADE.

REPAIRS: THE TOP 13-FT SECTION OF THE 32-IN OD COLUMN WAS REPLACED USING 1/4-IN 316L S.S. THE TRAYS AND DOWNCOMER 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 NAPHTHENIC ACID CORROSION. SEE I.N. 302 FOR FURTHER INFORMATION.
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 300. 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 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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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 APPROX. 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.
I.N.  PROCESS SEARCH CRITERIA
236  HYGAS  BELLOWS

TELECON, 4/27/76, FROM S. GREENBERG, ANL, TO B. ORCHARD, IGT.
EXPANSION JOINT-GASIFIER TRANSFER LINE.

SUMMARY

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.

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I.N.  PROCESS SEARCH CRITERIA
210  HYGAS  BELLOWS

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

SUMMARY

COAL PRODUCT-METAL REACTION PRODUCTS ARE FOUND GENERALLY ON BOTH SIDES OF THE BELLOWS AND IN ALL AREAS. IT SEEMS UNLIKELY THAT CORROSION IS THE MAIN CAUSE OF BELLOWS PERFORATIONS.

CURRENT VIEWS ARE THAT THE PITS ARE DUE TO EROSION, AT LEAST IN THE CASE OF INTERNAL SURFACE PITS. PITTING WAS PRECEDED BY LOCALIZED FAILURE OF THE LINER TUBE ALLOWING PARTICLE IMPINGEMENT ON THE INTERIOR BELLOWS SURFACE. AFTER PERFORATION OF THE BELLOWS THE STRUCTURE OF THE TRANSFER LINE ASSEMBLY ALLOWED PARTICLE IMPINGEMENT ON THE EXTERIOR SURFACE OF THE BELLOWS.

IT IS IMPORTANT TO EXAMINE THE REMAINDER OF THE BELLOWS AND ESPECIALLY THE LINER TUBE.

SEE I.N. 186, 194, 236, 274, 285, 286 FOR FURTHER INFORMATION.
I.N.  PROCESS  SEARCH CRITERIA
205  HYGAS  BELLOWS

ERDA MAT. AND COMP. FAIL. REPORT, 5/7/76, FROM W. ORCHARD, IGT.
3rd EXPANSION JOINT FROM LINE 322

SUMMARY

FAILURE: TWO LEAKS IN BELLOWS—LARGE LONGITUDINAL AND CIRCUMFERENTIAL CRACKS IN SHORT NIPPLE.
SERVICE LIFE: APPROX. 2 YRS INTERMITTENT SERVICE.
MATERIAL: INCOLOY 800
PRIOR REPAIRS: END NIPPLES CHANGED FROM TP446 TO INCOLOY 800 PRIOR TO INSTALLATION.
ENVIRONMENT: EXTERNAL—N2 AT 120F. INTERNAL—SYNTHESIS GAS AT 1200F (72% H2O, 9% H2, 9% CH4, 4% CO, 3% N2, < 1% H2S).
COMMENTS: CRACKS IN END NIPPLE APPEAR TO BE LOCATED AT THE SAME POINT WHERE THE LINER WAS ATTACHED TO THE END NIPPLE WITH A FILLET WELD.
ACTION: ANALYSIS TO BE PERFORMED BY ANL.

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I.N.  PROCESS  SEARCH CRITERIA
286  HYGAS  BELLOWS

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

SUMMARY

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.
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. 194, 210, 236, 274, 285, 286 FOR FURTHER INFORMATION.


STRESSES IMPORTANCE OF MAKING SURE THEY GET MATERIALS THEY SPECIFY AND ALSO THE IMPORTANCE OF CONTROLLING IN-PLANT WELDING PROCEDURES INCLUDING SPECIFYING OF TYPE OF WELD ROD.
FAILURE: DURING START-UP PRESSURE TESTING, LEAKING GAS WAS OBSERVED NEAR THE BOTTOM OF THE BELLOWS. EXACT LOCATION COULD NOT BE IDENTIFIED.

SERVICE LIFE: 8 MONTHS INTERMITTENT SERVICE.

ENVIRONMENT: EXTERNAL-N2 (ALSO INTERMITTENTLY HYDRAULIC OIL, WATER, COAL DUST) INTERNAL-650 F (72% H2O, 9% CH4, 4% CO, 3% CO2, 3% N2, < 1% H2S).

MATERIAL: INCOLOY 800 20 GAUGE.

CAUSE: EITHER PINHOLE LEAKS IN BELLOWS OR BELLOW SEPARATION FROM END PLATE IS THE EXPECTED CAUSE OF FAILURE.

ACTION: RETURNED TO MANUFACTURER.
EXPANSION JOINT-GASIFIER TRANSFER LINE AND AAG DIPLEG.

SUMMARY


STRESSES IMPORTANCE OF MAKING SURE THEY GET MATERIALS THEY SPECIFY AND ALSO THE IMPORTANCE OF CONTROLLING IN-PLANT WELDING PROCEDURES INCLUDING SPECIFYING OF TYPE OF WELD ROD.

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SUMMARY


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. BED TEMPERATURE IS 1850-1975F 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.

28
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.
I.N. PROCESS SEARCH CRITERIA
89 CO2 PIPING

INTEROFFICE REPORT, 8/7/72, FROM H. CROWDER TO J. LETERLE, CONOCO.
METALLOGRAPHIC STUDY OF 4-INCH DIAMETER INCOLOY 800 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.

I.N. PROCESS SEARCH CRITERIA
88 CO2 PIPING

FAILURE REPORT BY L. WOLFE, CONOCO, 8/14/72
METALLURGICAL STUDIES OF A RUPTURED INCOLOY 800 TUBE.

SUMMARY

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.

LETTER REPORT, 4/9/74, FROM L. RICE, C. F. BRAUN, TO D. GLASER, STEARNS-ROGER.
RISER EXPANSION JOINT FAILURES.

SUMMARY

REMEDIAL RECOMMENDATIONS FOR PIPING EXPANSION JOINT SPECIALISTS.
1. WEAKNESSES OF PRESENT SLIP JOINT SET-UP.
   A. PRESENT LOCATION WILL NOT PREVENT "JACK-KNIFING".
   B. ANY BINDING, SEIZING OR FRICTION AT THE JOINT WILL CAUSE BUCKLING AND
      DISTORTION OF THE TUBE DURING HEAT-UP AND SHUTDOWN.
2. CHANGES RECOMMENDED
   A. THREE DESIGN SKETCHES ARE ATTACHED SHOWING PROPOSED MODIFICATIONS.
   B. RECOMMEND THAT SLIP SLEEVES BE PURGED DOWNWARD RATHER THAN UPWARD
      AS AT PRESENT. THIS WILL PREVENT SOLIDS FROM ENTERING THE SLEEVE.
I.N. PROCESS SEARCH CRITERIA
95 CO2 PIPING

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.

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.
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 900 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, 3/25/75, FROM C. SCHULZ, CO2.
8-205 ACCEPTOR LIFT HEATER

SUMMARY

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. 1100-1400F. 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.
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. 1000-1500F, INERT GAS (83-87% N2, 6-12% CO2, 0.2-9.6% O2) AND RECYCLE GAS (30% 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: 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'-0" 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.
METALLURGICAL ANALYSIS OF INCOLOY 800 TUBE FROM B-201-1A HEATER COIL AFTER RUNS 20-22. FOLLOW-UP TO PREVIOUS LOCKHEED REPORT (LMS-0107190). 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.

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FAILURE: PIPE HAS ALMOST 360 DEGREE CIRCUMFERENTIAL BREAK IMMEDIATELY
ADJACENT TO O.D. WELD OF UPPER FLANGE, ALSO SOME LONGITUDINAL CRACKS ON
I.D. OF PIPE INSIDE SAME FLANGE.

SERVICE LIFE: 1-1/2 TO 2 YEARS OF INTERMITTENT SERVICE.

ENVIRONMENT: CHAR PLUS SYNTHESIS GAS AT ABOUT 1500 F (72% H2O, 9% CH4, 4% CO,
3% CO2, 3% N2, < 1% H2S).

MATERIALS OF CONSTRUCTION: 58" LENGTH OF 3" SCH. 40 PIPE (RA 330 OR INCOLOY 907)
EACH END HAVING A 150 LB 316 S.S. SLIP-ON FLANGE (WELDED WITH 312 OR 182 ROD).

CAUSE: BENDING FATIGUE. LOWER END OF THIS VERTICAL PIPE HANGS FREE INSIDE
FLUIDIZED BED.

ACTION: REPLACED PART. ANALYSIS BY ARGONNE NATIONAL LAB.
LETTER, 5/12/76, FROM D. SEVERSON, PROJECT LIGNITE, TO N. BERLIN, RALPH PARSONS.
PERSONNEL PROTECTION—PROJECT LIGNITE

SUMMARY

EXAMPLES OF SAFETY MEASURES UNDERTAKEN AT PROJECT LIGNITE FOR PERSONNEL
PROTECTION. THE SINGLE MOST SERIOUS POTENTIAL HAZARD IS CO WHICH IS BOTH TOXIC
AND FLAMMABLE. METHODS OF DEALING WITH THIS PROBLEM ARE DISCUSSED.
SECTIONS OF INCOLOY 800 TUBING FROM THE PDU HAVE BEEN EXAMINED PERIODICALLY
AND TO DATE (5/12/76) THERE HAVE BEEN NO INDICATIONS OF THE SCC THAT PLAGUED
THE 316 S.S. TUBING.
SEE I.N. 215 AND 225 FOR ADDITIONAL COMMENTS.
TRIP REPORT, 2/16/77, FROM R. KING & R. COOPER TO G. SLAUGHTER, ORNL.
REPORT ON TRIP TO SRC PILOT PLANT AT TACOMA, WASH.

SUMMARY

SRC PILOT PLANT PROBLEM AREAS

A. SLURRY PREHEATER COIL
1. > 20 FT HIGH X 10 FT DIAM., 1300-1700 FT LONG, 3" X SCHEDULE XX INCOLOY 800 HELICAL COIL, WITH BUTT WELDS.
2. PROBLEM IS AMOUNT OF COKING THAT OCCURS IN THIS COIL. IS IT SIGNIFICANT?
3. ORNL WILL TRY TO DEVELOP NOT METHODS FOR TESTING FOR COKING, CORROSION AND STRESS CORROSION IN THE COIL.

B. PARTIALLY COKED ELBOW
1. 5-D/4" SCHED. XX 347 S.S. (COLD FORMED NOT STRESS RELIEVED) ELBOW.
2. OPERATING TEMP. 750-800 F. 90% PLUGGED BY A HARD RESIDUE.
3. IF ELBOW PLUGS THE PROCESS IS SHUTDOWN. ONE PIPE HAS RuptURE DUE TO AUTOMATIC HEATING OF A PLUGGED PRESSURIZED LINE.
4. ORNL HAS A SECTION OF PLUGGED ELBOW FOR ANALYSIS.

C. DISSOLVER
1. A 347 S.S. LINER IN THE NOZZLE, WELDED TO A FLANGE THAT IS ATTACHED TO THE OVERLAY.
2. THE LINER-FLANGE WELD HAS CRACKED. POSSIBILITY EXISTS OF CORROSIVE ATTACK BENEATH THE SLEEVE.
3. ORNL WILL SUPPLY NDE GUIDANCE AND POSSIBLY FIELD EXAMINATION.

D. PRESSURE LETDOWN VALVES
1. 1" FISCHER VALVES (1980 AND ORGA) WITH FLOW FROM LARGE TO SMALL DIAMETER. TRIM SETS ARE TUNGSTEN CARBIDE.
2. VALVES ARE FAILING BY EROSION AND BREAKAGE FROM LODGED PARTICLES.
3. ORNL HAS RECOMMENDED SOME DESIGN CHANGES.
4. FAILED TRIM SETS WILL BE ANALYZED BY ORNL.

E. CENTRIFUGAL PUMPS
1. DIERKO PUMPS WITH CD4MCO HOUSINGS AND GOULD PUMPS WITH 12CR-HARDENED STEEL CASES. REBUILT AND FLAME SPRAYED WITH TUNGSTEN CARBIDE.
2. EROSION/CORROSION FAILURES OCCUR IN APPROX. 33 DAYS.
3. ORNL STRONGLY RECOMMENDS A PROGRAM FOR EVALUATING WEAR RESISTANT MATERIALS DIRECTLY IN THE SPC PROCESS STREAM. SRC MANAGEMENT WOULD PROBABLY SUPPORT THIS PROPOSAL.

F. PRESSURE VESSELS
1. HIGH PRESSURE DRUM (1500 PSI) - 2-1/4CR-1MO STEEL WITH A 2 FT I.D. X 5/6" THICK HEAD HAS A FORGE BONDED 304 S.S. CLAD.
2. STRESS CORROSION CRACKING OF HEAD IS PROBABLE.
3. INTERMEDIATE PRESSURE VESSEL (500 PSI) - 1/2MO STEEL 2 FT I.D. X 5/6"W.T.
4. CRACKING OF HEAD CLADDING IS VISIBLE.
5. ORNL COULD USE NDE TO DETERMINE ATTACK OF BASE METAL. NEXT SHUTDOWN THE HEAD COULD BE SECTIONED FOR COMPLETE ANALYSIS.

G. FRACTIONATION AREA
1. PROCESS STREAM IS HIGHLY ACIDIC WITH NAPHTHALIC ACID ATTACK AT ALL HOT AREAS BEING THE RULE. LOW ALLOY STEELS AND 304 S.S. ARE ATTACKED.
CRACKING IN INCOLOY 800 PIPE REDUCER

SUMMARY

FAILURE: NOZZLE NO. 50 DEVELOPED CRACKS IN THREE AREAS AROUND THE WELD. CRACKS APPEAR TO BE IN HAZ WHERE MONEL LINER IS WELDED TO INSIDE OF REDUCER.

SERVICE LIFE: SINCE INITIAL START-UP.

ENVIRONMENT: 600 PSIG, SKIN TEMPERATURE, 300-400 F., DOAL PRODUCT GAS.

MATERIAL: NOZZLE IS 3" IN DIAM. AND REDUCES TO 1-1/2" PIPE AND FLANGE. INCOLOY 800 LINED WITH MONEL OVERLAY.

CAUSE: UNKNOWN. REPORT DUE LATER.

ACTION: FAULTY WELD AREA HAS BEEN CUT OUT AND WILL BE REPLACED BY A 3" PIPE SECTION WITHOUT THE REDUCER.

SEE I.N. 410 FOR FURTHER ANALYSIS OF FAILURE.

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CRACKING IN AN INCOLOY 300 REDUCEP-SYNTHANE PILOT PLANT

SUMMARY

SEE I.N. 405 FOR INITIAL FAILURE REPORT.

FAILURE MODE: METALLOGRAPHIC EXAMINATION BY C.E. LUMMUS INDICATED THAT INTERGRANULAR STRESS CORROSION CRACKING WAS THE CAUSE OF FAILURE. IN ADDITION, THE AS-RECEIVED INCOLOY 300 BASE METAL WAS SENSITIZED.

RECOMMENDATIONS

1. REMOVE NOZZLES 39 AND 41 INCLUDING FLANGES AND RING JOINTS FOR EXAMINATION BY LUMMUS.
2. INITIATE REPLACEMENT OF NOZZLES USING MONEL-LINED COMPONENTS IN COMBINATION WITH ON-HAND INCOLOY COMPONENTS. ALL INCOLOY 300 USED WILL BE SOLUTION ANNEALED AND QUENCHED.
3. THIS WILL REQUIRE A SHUTDOWN OF APPROXIMATELY 5 WEEKS. DURING THIS TIME VARIOUS OTHER MAINTENANCE AND REPAIR TASKS WILL BE COMPLETED.
FAILURE: 1/32" DIAM. HOLE DEVELOPED IN "Y" BRANCH OF DOLOMITE DIVERSION PIPE LINE.
SERVICE LIFE: 30 HRS.
ENVIRONMENT: 1200 F. DEAD BURNED DOLOMITE. 240 PSIG.
MATERIAL: 1/2-INCH SCHEDULE 40 INCOLOY 800 PIPE.
CAUSE: 45 DEGREE "Y" BRANCH INTERNALLY ERODED DUE TO ABRASION OF MATERIAL.
ACTION: REPLACED PART. ANGLE OF BRANCH CHANGED TO 90 DEGREES IN ORDER TO REDUCE IMPACT VELOCITIES. ANALYSIS TO BE DONE BY COMPANY LABS.

FAILURE: CRACK IN PIPE 1/32" WIDE, 2/3 OF CIRCUMFERENCE, 7/8" FROM FLANGE TO FI114 HEAT EP.
SERVICE LIFE: 2 YEARS.
ENVIRONMENT: 525 F. MAX. RECIRCULATING GAS.
MATERIAL OF CONSTRUCTION: INCOLOY 300. 1" DIAM. PIPE.
CAUSE: FAILURE IN HAZ NEAP WELD. MATERIAL FLAW. FAILURE CAUSED BY VIBRATION INDUCED CRACK PROPAGATION.
ACTION: REPLACED PART. SAVED. NO ANALYSIS.
SEE I.N. 409 FOR ANALYSIS OF PIPING FAILURE.
PROCESS   SEARCH CRITERIA
WESTINGHOUSE   PIPING

MEMO, 6/21/77, FROM T. COX TO M. CARRINGTON, ERDA
CRACKING OF INCOLOY 800 PIPING IN WESTINGHOUSE PDU

SUMMARY

FAILURE: SIX FAILURES HAVE OCCURRED IN THE 1-INCH, SCHEDULE 40 PIPE IN THE
TRANSPORT LINES. FOUR FAILURES HAVE OCCURRED IN THE 4-INCH, SCHEDULE 40 PIPE IN THE TRANSPORT LINES. FOUR FAILURES HAVE OCCURRED IN THE 2-INCH SCHEDULE 40 PIPE IN THE VENT PIPING. FAILURE CAUSE WAS CRACKING WHICH OCCURRED CLOSE TO WELDS, BUT NOT NECESSARILY IN THE WELD METAL OR HAZ.
SERVICE LIFE: FAILURES OCCURRED OVER A PERIOD OF SEVERAL HUNDRED HOURS OF OPERATION.
ENVIRONMENT: MAXIMUM TEMPERATURE IS 600F.
MATERIAL: INCOLOY 300
CAUSE: EVIDENCE INDICATES THAT THE FAILURES RESULTED FROM INTERGRANULAR STRESS CORROSION CRACKING. EXAMINATION REVEALED THAT THE INCOLOY 800 WAS HIGHLY SENSITIZED. RESIDUAL STRESSES MAY BE DUE TO THE WELDING OPERATION OR TO IMPOSED STRESSES DURING INSTALLATION.
THE CORRODING AGENT HAS NOT BEEN IDENTIFIED.

RECOMMENDATIONS
1. SENSITIZED PIPING MUST BE ELIMINATED.
2. REPLACE WITH SOLUTION ANNEALED AND QUENCHED MATERIAL WELDED USING A PROCEDURE TO AVOID SENSITIZATION IN THE HAZ.
3. EFFORTS SHOULD BE MADE TO IDENTIFY THE CORRODING AGENT.
4. COLLECTION POINTS SHOULD BE INSTALLED AT SUITABLE POSITIONS IN THE PDU FOR OBTAINING SAMPLES OF THE CONDENSATES WHICH FORM IN THE LINES.
SEE I.N. 353 FOR INITIAL REPORT OF ONE OF THE PIPING FAILURES.

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PRESSURE VESSELS
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 REDISEGN 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. 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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I.N. PROCESS SEARCH CRITERIA
258 SYNTANHE PRESSURE VESSEL

ERDA MAT. AND COMP. FAIL. REPORT, 10/18/76, FROM J. JEWELL, BRUCETON.
GASIFIER SHROUD

SUMMARY

FAILURE: GASIFIER SHROUD BUCKLED
SERVICE LIFE: O2 IN 94 HRS TO DATE, 18-1/2 HRS ON LAST RUN
ENVIRONMENT: HS STEAM, O2, COAL AT 1500F, RUN M.
MATERIAL: 1/8" THICK INCOLOY 800H (SB-409-900 GR 2 ANNEALED)
CAUSE: EQUIPMENT MALFUNCTION, OVERHEATING FAILURE.
ACTION: REPLACED SHROUD, REPORT EXPECTED BY 1/2/77.

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I.N. PROCESS SEARCH CRITERIA
377 SYNTANHE PRESSURE VESSEL

QUALITY ASSURANCE REPORT, 4/23/77, BY J. JEWELL, LUMMUS.
BUCKLED GASIFIER SHROUD

SUMMARY

FAILURE: CRACKS WERE FOUND IN THE WELD BETWEEN THE INTERFACE WITH THE CONE.
SERVICE LIFE: 450 HRS OF OXYGEN.
ENVIRONMENT: COAL DUST, OXYGEN, STEAM, 600 PSIG, 1500 F.
PRIOR REPAIR: SHROUD HAS BUCKLED PREVIOUSLY.
MATERIAL: INCOLOY 800
CAUSE: INTERNAL STRESS AND THERMAL STRESSES CAUSED BY BUCKLED SHROUD LED TO WELD FAILURE.
ACTION: PRESS SHROUD BACK INTO POSITION ANDREWELD.
COMMENT: THOUGHT SHOULD BE GIVEN TO A NEW DESIGN, ONE WITH LOOSE PLATES, TO ALLOW FOR EXPANSION AND CONTRACTION. PRESENT DESIGN ALLOWS NO ROOM FOR EXPANSION.
INTEROFFICE MEMO, 5/9/77, FROM W. V. VAN SWERINGEN TO P. STREIBINGER, LUMMUS.
CRACKING IN GASIFIER SHROUDS

SUMMARY

CRACKS HAVE APPEARED IN THE CYLINDRICAL INCOLOY 800 MANWAY SHROUDS ON THE 7TH AND 10TH FLOORS. THESE CRACKS RUN CIRCUMFERENTIALLY FROM 8:00 TO 4:00 AND ARE APPROX. 3/4" AWAY FROM THE GASIFIER SHELL. THERE ARE SEVERAL SHORT AXIAL CRACKS BRANCHING FROM THE MAIN CRACK POSSIBLY CAUSED BY ATTEMPTED WELD REPAIRS.

THE CRACKING WAS PROBABLY CAUSED BY THE SEVERE THERMAL GRADIENT THAT EXISTS IN THE SHROUD (APPROX. 1400-200 F OVER 9") BETWEEN THE INSIDE OF THE CASTABLE AND THE SHELL. ANOTHER HYPOTHESIS IS THAT CRACKING OCCURRED FROM A COMBINATION OF EFFECTS—DIRECT THERMAL STRESSES, CYCLIC HEATING AND COOLING, AND VIBRATION FATIGUE.

IT WAS CONCLUDED THAT THE CRACKS WILL DO LITTLE HARM. 3/8" HOLES WILL BE DRILLED AT THE END OF EACH CRACK TO STOP ITS PROPAGATION.

SEE I.N. 377, 393, 399 FOR ADDITIONAL INFORMATION.

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QUALITY ASSURANCE REPORT, 5/13/77, FROM J. JEWELL, LUMMUS.
EROSION OF GASIFIER CYCLONE

SUMMARY

FAILURE: HOLE FOUND IN CONE SECTION OF CYCLONE. AFTER REMOVAL OF THE CYCLONE A GENERAL EROSION PATTERN WAS FOUND.
SERVICE LIFE: APPROX. 1000 HRS.
ENVIRONMENT: 800 F, PRODUCT GAS, CHAR AND COAL DUST, 600 PSIG.
MATERIAL: INCOLOY 800.
CAUSE: EROSION.
ACTION: REPAIRED BY ADDING TWO CONICAL COLLARS 1/8" THICK OVER ENTIRE CONE AREA.
QUALITY ASSURANCE REPORT, 5/13/77, FROM R. LORD, LUMMUS.
CRACK IN GASIFIER SHROUD

SUMMARY

FAILURE: CRACK IN SHROUDING IN 7TH FLOOR MANWAY FROM 8:00 TO 4:00 POSITION.
CRACK NEAR SHROUD TO SHELL WELD ABOUT 1/8" WIDE.
SERVICE LIFE: UNKNOWN.
ENVIRONMENT: UNKNOWN.
MATERIAL: INCOLOY 800.
CAUSE: APPARENT CAUSE OF FAILURE WAS THERMAL STRESSES.
ACTION: LEAVE AS IS FOR NEXT RUN, REPAIR, AND INSTALL NEW REFRACTORY DURING NEXT SHUTDOWN.
COMMENTS: 3/16" HOLES WERE DRILLED AT THE ENDS OF THE CRACKS THROUGH THE SHROUD TO PREVENT FURTHER CRACKING.
SEE I.N. 377, 393 FOR PREVIOUS REPORTS ON THIS PROBLEM.

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QUALITY ASSURANCE REPORT, 5/24/77, FROM J. JEWELL, LUMMUS.
GASIFIER—BUCKLED SHROUD

SUMMARY

FAILURE: THE INNER SHROUD IN THE REACTION ZONE HAS BEEN BUCKLED FOR SOME TIME. IT IS BELIEVED THAT THE BULGES INTERFERE WITH THE FLUIDIZED BED AND POSSIBLY CAUSE THE FORMATION OF GLINKERS.
SERVICE LIFE: 400 HRS.
ENVIRONMENT: 1500 F, 600 PSIG, COAL GAS, O2, STEAM, COAL CHAR.
MATERIAL: INCOLOY 900.
CAUSE: PREVIOUS REPORT INDICATES THAT A POOR DESIGN WHICH FAILS TO ALLOW FOR THERMAL EXPANSION IS THE CONTRIBUTING FACTOR. SEE I.N. 377.
ACTION: REMOVED 90% OF ALL SHROUD, WELDED IN NEW SHROUD 360 DEGREES AS SHOWN BY PHOTO. NO MENTION MADE OF DESIGN CHANGE.
THERMOCOUPLES
PRELIMINARY REPORT, 7/26/76, FROM D. DUBIS TO B. LEWIS, SYNTHANE.
GASIFIER DISTRIBUTOR CONE THERMOCOUPLE FAILURE.

SUMMARY

SUMMARY OF PRELIMINARY REPORT FROM S. DANYLUK, ANL.
1. THE FAILURE APPEARS TO BE CAUSED BY INTERGRANULAR CORROSION. THE CRACK STARTS AT OUTSIDE WALL AND PROGRESSES INWARD.
2. THIS FORM OF CRACKING IS AGGRAVATED BY A HIGH SULFUR ENVIRONMENT.
3. MGO IS HYDROSCOPIC AND THE EXPANDING MOISTURE WILL COMPLETE THE TUBE SPLIT.
4. USE 310 OR 330 S.S. FOR BETTER CORROSION RESISTANCE, DEFINITELY NOT INCOLOY 800.
5. CHECK ALL NEWLY INSTALLED THERMOCOUPLES WITH MAGNIFYING GLASS FOR CRACKS.
6. ANL PLANS TO DO CHEMICAL ANALYSIS OF SHEATH MATERIAL, MGO AND CORROSION PRODUCT.

SEE I.N. 295, 298, 299, 300, 307, 308 FOR FURTHER INFORMATION.

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I.N. PROCESS SEARCH CRITERIA

300 SYNTHANE THERMOCOUPLES

TELECON, 8/2/76, FROM R. IVINS, ANL, TO B. ROCHE, LUMMUS.
FAILURE ANALYSIS OF DISTRIBUTION CONE THERMOCOUPLE SHEATHS.

SUMMARY

ANL HAS CONTACTED THERMOCOUPLE SUPPLIER AND HOPES TO OBTAIN SAMPLES FROM SAME BATCH AS FAILED THERMOCOUPLES.
G. STANTON IS TO BRING BACK SAMPLES FROM A SECOND BATCH OF FAILED T/C SHEATHS.

SEE I.N. 295, 298, 299, 303, 307, 308 FOR FURTHER INFORMATION.
PROCESS
SEARCH CRITERIA

I.N. 308  SYNTHANE  THERMOCOUPLES

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.

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I.N. 319  SYNTHANE  THERMOCOUPLES

TELECON, 9/3/76, FROM S. DANYLUK, ANL, TO B. LEWIS AND D. BAILEY, SYNTHANE.
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 0.25% 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.
FAILURE: Corrosion cracks initiated at the OD of the sheath material at surface cracks or pits. The cracks penetrated to the ID and the \( \text{MGO} \) was exposed to steam. These cracks widened because of formation of \( \text{MGO(OH)}_2 \) which has double the molecular volume of \( \text{MGO} \). 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 650°F.

MATERIALS OF CONSTRUCTION: CR-AL thermocouples with a sheath of Incoloy 800 and \( \text{MGO} \) 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.
LETTER W/COPY OF TC STANDARDS, 11/4/74, FROM S. DANYLUK, ANL, TO B. ROCHE, LUMMUS.
RDT 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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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 AVAILABLE FOR SEVERAL MONTHS. 446 S.S. WAS RECOMMENDED BUT EMBRITTLEMENT
MAY BE A PROBLEM.
VALVES
FAILURE: THE LINER OF THE VALVE WAS ERODED AWAY IN A SECTION 3" WIDE ON
THE BOTTOM TO 1" WIDE ON THE TOP ON ONE SIDE OF THE VALVE.
SERVICE LIFE: APPROX. 9 MONTHS.
ENVIRONMENT: TEMP. 145OF. VELOCITY 55-100 FT/SEC. ENTRAINED DOLOMITE
8 X 20 MESH, RECYCLE GAS (CO, CO2, N2) WITH SULFUR IN THE PPM RANGE.
MATERIAL OF CONSTRUCTION: INCOLOY 800
CAUSE OF FAILURE: THE INNER LINER WAS MISALIGNED DURING MANUFACTURE CAUSING
EROSION ON THE LINER.
ACTION: THE REPLACEMENT VALVE INNER LINER WAS PROPERLY ALIGNED.

*      *      *      *      *
**Report and Subtitle:**
Report of Coal Conversion Plants Operating Experiences
Materials of Construction - Incoloy 800

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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 use of Incoloy 800 in coal conversion processes.

**Key Words:**
Coal gasification; coal liquefaction; component failures; failure analysis; Incoloy 800; material evaluation

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Unlimited

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