CORROSION CONTROL
ANNUAL RESEARCH REVIEW
HANDOUT BOOK

April 2, 1991

Atlanta, Georgia
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FUNDAMENTALS OF CORROSION CONTROL IN PAPER MILLS
CORROSION CONTROL

ANNUAL RESEARCH REVIEW
INTRODUCTION

- PAPER MACHINE CORROSION
- CHEMICAL RECOVERY BOILER CORROSION
PAPER MACHINE CORROSION

- SUCTION ROLL CRACKING

- ELECTROCHEMICAL STUDIES IN WHITE WATER
RECOVERY BOILER CORROSION

• LOWER FURNACE
  - GAS
  - SMELT

• PORT CORROSION
  - ALKALI HYDROXIDE
  - STAINLESS STEEL
PAPER MACHINE CORROSION

- CORROSION FATIGUE FAILURES ARE COSTLY
  - Mechanism
  - Test Development

- TEST DEVELOPMENT GOALS
  - Alloy Ranking Consistent with Service
  - Develop New Alloys (Mechanism)
FUNDAMENTALS OF PAPER MACHINE CORROSION

TASK 1
Fatigue in Concentrated Chloride Solutions

TASK 2
Mean Stress Effects

TASK 3
Microstructural Analysis

TASK 4
Crack Propagation Rates
<table>
<thead>
<tr>
<th>Alloy</th>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>CA-15</td>
<td>0.7</td>
<td>12.5</td>
<td>0.5</td>
<td>2.5</td>
<td>---</td>
</tr>
<tr>
<td>A-63</td>
<td>0.5</td>
<td>22</td>
<td>9.5</td>
<td>7</td>
<td>---</td>
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<tr>
<td>A-75</td>
<td>0.02</td>
<td>26</td>
<td>6</td>
<td>6</td>
<td>2.8</td>
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<tr>
<td>A-86</td>
<td>0.02</td>
<td>26</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
PRIOR RESULTS

- CRACK INITIATION
  - S-N Curves

- CRACK PROPAGATION
  - Near-threshold Rates
  - Threshold

- ENVIRONMENTAL VARIABLES
  - Chloride, Sulfate, Thiosulfate

- MEAN STRESS
  - R-ratio
TASK 1 OBJECTIVES

• INVESTIGATE PIT ACIDIFICATION EFFECT ON CRACK INITIATION

• EVALUATE ROTATING BENDING METHOD FOR FUTURE APPLICATION
TASK 1 PROCEDURES

- ALLOYS A63 and A75
- Fe, Cr, Ni CHLORIDES
- ELECTROCHEMICAL TESTS
  - 4 M and 10 M Cl-
- FATIGUE TESTS
  - Rotating Bending
  - 2 M and 6 M Cl-
S-N Curves for Alloys A63 and A75 in Concentrated Chloride Solutions (After Huhn).
TASK 1 CONCLUSIONS

• A63 HAS BETTER LOCALIZED CORROSION RESISTANCE THAN A75

• FATIGUE BEHAVIOR IN REVERSED BENDING DOES NOT AGREE WITH SERVICE

• INHERENT CORROSION RESISTANCE IS NOT CRITICAL FOR CORROSION FATIGUE OF DUPLEX STAINLESS STEELS
TASK 2 OBJECTIVES

- SIMULATE RESIDUAL STRESS EFFECT
  - Alloy Processing
  - Weld Repairs

- DEVELOP DATA PRESENTATION TECHNIQUE
TASK 2 PROCEDURES

- UNIAXIAL TENSION
- SMOOTH SPECIMENS
- CHLORIDE, SULFATE AND THIOSULFATE
- $T = 50 \, ^{\circ}C$
- $pH = 3.5$
TASK 1 RESULTS

- **ANODIC POLARIZATION; 4 M Cl-**
  - A63: superficial pitting
  - A75: severe pitting

- **ANODIC POLARIZATION; 10 M Cl-**
  - A63: limited uniform corrosion
  - A75: severe uniform corrosion

- **2 M and 6 M Cl- SELECTED FOR FATIGUE TESTS**
S-N Curve for A86 and CA-15.
Goodman Diagram for Alloys A86 and CA-15.
S-N Data for Alloys A63 and A75.
Rotating bending at pH of 3.5, Cl⁻ of 20 ppm.
(After Dahl)
Goodman Diagram for Alloys A63 and A75.
Data from Figure 5 at a lifetime of $5 \times 10^7$ cycles.
Optical micrograph of the surface of CA-15 showing pits. Note the one on the fracture surface.
Scanning Election Micrograph of CA-15 showing Depth of pit on fracture surface.
Scanning Electron Micrograph depicting multiple fatigue crack initiation sites in Alloy A86.
TASK 3 OBJECTIVES

- DEVELOP INFORMATION ON CRACK PATH IN DUPLEX STAINLESS STEELS
- CORRELATE WITH MICROSTRUCTURE
- RECOMMEND ALLOY DEVELOPMENT STRATEGY
TASK 3 PROCEDURES

- METALLOGRAPHIC
  - Optical
  - SEM

- INVESTIGATE FRASTA
  - Computer Reconstruction
  - Correlate with Microstructure
TASK 4 OBJECTIVES

- DETERMINE IF CRACK PROPAGATION CAN BE USED TO RANK ALLOYS
  - Threshold
  - Near-threshold Crack Propagation Rate
TASK 4 PROCEDURES

- PRECRACKED COMPACT TENSION SPECIMENS
- A75 AND A86
- CHLORIDE, SULFATE AND THIOSULFATE
- T = 50 C
- pH = 3.5
Corrosion Fatigue Crack growth: A86 at R=0.1

ALL ENVIRONMENTS
pH = 3.5
T = 50 C
f = 25 Hz

Fatigue Crack Propagation of Alloy A86 in Three Different White Waters.
Fatigue data: A75 vs A86

$\Delta K$ (Ksi/$\sqrt{\text{in}}$)

Comparison of Fatigue Crack Propagation Rates for Alloys A75 and A86 at $R = 0.1$.
Fatigue data: A75 vs A86

Comparison of Fatigue Crack Propagation Rates for Alloys A75 and A86 at R = 0.5.

- A86 R = 0.5
- A75 R = 0.5

1000 ppm Cl, pH = 3.5, T = 50 C, f = 25 Hz
Corrosion-fatigue: A75 vs A86

Fatigue Crack Propagation Rates of Alloys A75 and A86 in a Simulated White Water.
Table 1. Threshold Stress Intensities for A75 and A86 for 1000 mg/l Cl⁻ at a pH of 3.5 and Temperature of 50°C.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>R-ratio</th>
<th>ΔKₚ (ksi_in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A75</td>
<td>0.1</td>
<td>10.2</td>
</tr>
<tr>
<td>A86</td>
<td>0.1</td>
<td>10.1</td>
</tr>
<tr>
<td>A75</td>
<td>0.5</td>
<td>6.9</td>
</tr>
<tr>
<td>A86</td>
<td>0.5</td>
<td>5.7</td>
</tr>
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</table>
Table 2. Threshold Stress Intensities for A75 and A86 for 200mg/l Cl, 500mg/l SO\textsubscript{4}\textsuperscript{2-} and 50mg/l S\textsubscript{2}O\textsubscript{3}\textsuperscript{2-} at a pH of 3.5 and Temperature of 50°C.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>R-ratio</th>
<th>$\Delta K_{th}$ (ksi·in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A75</td>
<td>0.1</td>
<td>8.9</td>
</tr>
<tr>
<td>A86</td>
<td>0.1</td>
<td>10.6</td>
</tr>
</tbody>
</table>
Scanning Electron Micrograph of the Fracture Surface of A86.
TASK 4 RESULTS

- RANKING IS DEPENDENT ON ENVIRONMENT
- MICROSTRUCTURE MAY PLAY A LARGE ROLE
SUMMARY

• PIT ACIDIFICATION DOES NOT APPEAR TO CONTROL CRACK INITIATION

• GOODMAN DIAGRAMS MAY PROVE USEFUL IN ALLOWING RESIDUAL STRESS EFFECTS TO BE CONSIDERED
SUMMARY (cont.)

• MICROSTRUCTURAL ANALYSIS MAY PROVIDE CLUES TO CRACK PATH AND THE MECHANISM OF CRACK INITIATION

• CRACK PROPAGATION STUDIES DO NOT DISCRIMINATE BETWEEN DUPLEX STAINLESS STEELS
PROJECT 3534-42
ELECTROCHEMICAL STUDIES IN WHITE WATER
ELECTROCHEMICAL STUDIES IN
WHITE WATER

Project leader: Tero Hakkarainen

(visiting scientist from The Technical Research Centre of Finland, VTT)
OBJECTIVE

To relate the steel composition and microstructure to the susceptibility to pitting & crevice corrosion.

* of cast suction roll duplex stainless steels
* in white water with thiosulfate
* by electrochemical experiments and
* microstructural observations
BACKGROUND

* Laboratory corrosion fatigue tests vs. practical experience (suction roll stainless steels)

* Possible role of localized attack (pitting & crevice corrosion)

* Effects of thiosulfate on austenitic stainless steels
## TEST MATERIALS

<table>
<thead>
<tr>
<th>Alloy</th>
<th>%Cr</th>
<th>%Ni</th>
<th>%Mo</th>
<th>%Cu</th>
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</thead>
<tbody>
<tr>
<td>63</td>
<td>22</td>
<td>10</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>75</td>
<td>26</td>
<td>6.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>86</td>
<td>26</td>
<td>6.5</td>
<td>2</td>
<td></td>
</tr>
</tbody>
</table>

Alloys 75 and 86 slowly cooled

Alloy 63 water quenched
MICROSTRUCTURES

Alloy 75
* Approximately 60% austenite
* Dual appearance:
  - coarse dendrites
  - areas of fine distribution
* Small colonies presumably containing sigma phase

Alloy 86
* Very similar to Alloy 75

Alloy 63
* Homogeneous structure
  - no finely distributed austenite
  - no "sigma phase colonies"
Microstructure of Alloy 75. Optical micrographs.
a) General view, 100x.
b) Detail showing a "sigma phase" colony, 1000x.
Microstructure of Alloy 86. Optical micrographs.
a) General view, 100x.
b) Detail showing "sigma phase" colonies, 1000x.
Microstructure of Alloy 63. Optical micrograph. 100x.


**ENVIRONMENTS**

* Basic white water:

<table>
<thead>
<tr>
<th>Component</th>
<th>mg/l</th>
<th>mol/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>sulfate</td>
<td>500</td>
<td>0.0052</td>
</tr>
<tr>
<td>chloride</td>
<td>200</td>
<td>0.0056</td>
</tr>
<tr>
<td>thiosulfate</td>
<td>50</td>
<td>0.00045</td>
</tr>
</tbody>
</table>

50°C, pH = 3.5

* Only one or two of the components present
* Less chloride or/and thiosulfate
* More (less) sulfate
TEST PROCEDURES

Cyclic potentiodynamic "pitting tests"
- \(0.2 \text{ mV/s (12 mV/min)}\)
- reversal of scan direction at 1 mA

Potentiostatic tests
- basic white water
- increasing chloride & thiosulfate contents

Specimens: wet ground or polished & etched
Basic white water:

- 200 mg/l chloride
- 500 mg/l sulfate
- 50 mg/l thiosulfate

75 = Alloy 75
86 = Alloy 86
63 = Alloy 63
Alloy 75

A: 67 mg/l chloride
   500 mg/l sulfate
   50 mg/l thiosulfate

B: 200 mg/l chloride
   500 mg/l sulfate
   17 mg/l thiosulfate

C: 200 mg/l chloride
   1500 mg/l sulfate
   50 mg/l thiosulfate
Alloy 75

500 mg/l sulfate
50 mg/l thiosulfate

A: anodic current
B: cathodic current
Alloy 80

A: 200 mg/l chloride
B: 200 mg/l chloride
   50 mg/l thiosulfate
Alloy 75
500 mg/l sulfate
+ chloride
+ thiosulfate

-200 mV
Alloy 75  500 mg/l sulfate
+ chloride
+ thiosulfate

-200 mV
Alloy 75

-100 mV
Alloy 86  500 mg/l sulfate
200 mg/l chloride
50 mg/l thiosulfate

-200 mV:
A: wet ground
B: polished and etched
Pitting attack of Alloy 75 in the basic white water after 10 minutes at -200 mV(SCE). Specimen surface polished and etched prior to exposure. Pits covered with corrosion products. Optical micrographs. a) 200x, b) 500x.
Pitting attack of Alloy 86 in the basic white water after 55 minutes at -200 mV(SCE). Specimen surface polished and etched prior to exposure. Pits covered with corrosion products. Optical micrograph. 500x.
Pitting attack of Alloy 75 in the basic white water after 10 minutes at -200 mV(SCE). Specimen surface polished and etched prior to exposure and again after the exposure. Optical micrograph, 400x.
CONCLUSIONS

Alloy 75 is susceptible to thiosulfate induced pitting in white water,

Alloy 86 is marginally susceptible,

Alloy 63 is practically immune.

Initiation of attack at colonies containing sigma phase and austenite.

The colonies probably do not otherwise impair the corrosion resistance or the mechanical properties of the alloys.

(Continued...)

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CONCLUSIONS (Cont’d)

For thiosulfate attack to occur, both the electrode potential and the relative content of the thiosulfate ions have to be within certain ranges.

For rapid pit growth (Alloy 75), the presence of chloride ions is required.

If thiosulfate ions are present, localized attack induced by chloride ions at high potentials may continue at low potentials in Alloys 75 and 86, but probably not in Alloy 63.

(Continued...)

-65-
Copper (Alloy 86) strongly inhibits and molybdenum (Alloy 63) eliminates the thiosulfate pitting of duplex stainless steel (Alloy 75) in white water.

The differences in service performance (corrosion fatigue cracking) between Alloys 63 and 75 are not explained by differences in the susceptibilities to localized attack in white water.
FURTHER WORK

Scanning electron microscopy
- relation between attack and microstructure
- role of copper

Transmission electron microscopy
- identification of phases in colonies

"Avesta cell"
- threshold concentrations
PROJECT 3628
RECOVERY BOILER FIRESIDE CORROSION
Project Objective

TO UNDERSTAND THE CAUSES OF CORROSION ON THE FIRE-SIDE OF THE WATER WALL TUBES IN THE LOWER FURNACE OF KRAFT RECOVERY BOILERS AS BASIS FOR DEVISING METHODS OF REDUCING CORROSION DAMAGE.
Task 1., Port Corrosion

Objectives

1. IDENTIFY THE CORROSIVE AGENT(S)
   and determine the effect of:

2. ENVIRONMENTAL FACTORS

3. STEEL COMPOSITION

4. GALVANIC CONTACT
Overall Project Scope
Task 1., Port Corrosion
Experimental Approach
Electrochemical tests in molten NaOH
Polarization curves for carbon steel and stainless steel obtained simultaneously in molten NaOH under N₂ at 320 °C.
Polarization curves for carbon steel and stainless steel obtained simultaneously in molten NaOH under N₂ at 320
Polarization curves for carbon steel and stainless steel obtained simultaneously in molten NaOH exposed to air for 2.5 hours.
Polarization curves for carbon steel and stainless steel obtained simultaneously in molten NaOH exposed to air for 21 hours.
Task 1., Port Corrosion

Current Results

STAINLESS STEEL CORRODES PREFERENTIALLY IN NaOH MELT UNDER N₂ AND AIR WHEN IN CONTACT WITH CARBON STEEL.
Task 1., Port Corrosion
Experimental Approach (cont.)
Field testing with port probes

![Diagram of experimental setup]

- SS SPECIMEN
- Pt ELECTRODES
- CARTRIDGE HEATER
- T-CONTROLLER
- AIR IN
- wire leads
- AIR OUT
Task 1., Port Corrosion
Future Work

1. TESTS WITH CO₂ IN THE GAS OVER NaOH MELT
2. TESTS WITH H₂O IN THE GAS OVER NaOH MELT
3. TESTS WITH VARIOUS SS GRADES
4. FIELD TESTING
Task 2., Gas / Smelt Reactions

Objectives

1. STUDY OF:
   
   A) GAS REACTIONS
   B) REACTIONS BETWEEN GAS AND SMELT

2. THE EFFECT OF SMELT ADDITIVES ON CORROSION
Task 2.
Gas / Smelt Reactions
Experimental Approach

Horizontal tube furnace for corrosion tests with coupons in gas and gas + smelt powder environments
Carbon Steel, 5 hr

Corrosion Rate (mpy)

- NO
- KCl
- Pyro
- NaOH
- Sulfite
- NaCl
- S
- Thio

Smelt Additive (5%)

- 330 C
- 440 C

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Task 2., Gas/Smelt Reactions
Initial and Equilibrium Gas

init.: \[10\% \text{CO}_2 + 2.0\% \text{H}_2\text{O} + 1.0\% \text{H}_2\text{S} + 1\% \text{O}_2\]
eq 300: \[10\% \text{CO}_2 + 2.9\% \text{H}_2\text{O} + 0.56\% \text{SO}_2 + 0.16\% \text{H}_2\text{S} + 400 \text{ ppm S}_8 + 90 \text{ ppmS}_2\]
eq 400: \[10\% \text{CO}_2 + 2.8\% \text{H}_2\text{O} + 0.62\% \text{SO}_2 + 0.24\% \text{H}_2\text{S} + 660 \text{ ppm S}_2 + 3 \text{ ppmS}_8\]

at equilibrium: also traces of COS, H$_2$, CO, SO$_3$, and O$_2$

Main reaction:

\[\text{H}_2\text{S} + \text{O}_2 \Rightarrow \text{H}_2\text{O} + \frac{1}{2} \text{SO}_2 + \frac{1}{2} \text{S}_x\]
Task 2., Gas/Smelt Reactions

Reactions between gas and smelt

init. : 8.0% Na$_2$S, 12% Na$_2$SO$_4$, and 80 % Na$_2$CO$_3$,
after 5h: 0.2% Na$_2$S, 16% Na$_2$SO$_4$, and 40% Na$_2$CO$_3$

Thermodynamically feasible reactions:

Na$_2$S + CO$_2$ + H$_2$O $\rightarrow$ H$_2$S + Na$_2$CO$_3$
Na$_2$S + 2CO$_2$ $\rightarrow$ Na$_2$CO$_3$ + 1/2S$_2$ + CO
Na$_2$CO$_3$ + SO$_2$ + 1/2 O$_2$ $\rightarrow$ CO$_2$ + Na$_2$SO$_4$
Task 2., Gas / Smelt Reactions

Results and Conclusions

1. TEST GAS TENDS TO GENERATE $S_X$, $SO_2$, AND $H_2O$

2. THE GAS REACTS WITH SMELT AND MAY RELEASE $H_2S$ AND $CO_2$

3. SULFUR, THIOSULFATE, SULFITE OR PYROSULFATE ADDED TO SMELT AGGRAVATE THE CONDITION

4. CHLORIDE AND HYDROXIDE ADDITION INCREASE CORROSION

5. $H_2O$ SEEMS TO RETARD CORROSION
Task 2., Gas/Smelt Reactions

Future Work

STUDY OF:

1. H₂O-EFFECT ON CORROSION RATE
2. THE EFFECT OF SO₂ AND CO₂ ON SMELT
Task 3., Continuous Kinetics

Objectives

1. CORROSION RATE DEPENDENCY WITH TIME
2. SCALE MORPHOLOGY
RECOVERY BOILER CORROSION

Carbon Steel Coupons (C1018)

No Smelt Present

Equation of line
\[ w = -386 + 248(\ln t) \]
Slope = 4500/t (mpy)

63 Mpy

Instantaneous Corrosion Rate

Equation of line
\[ w = -18 + 17(\ln t) \]
Slope = 310/t (mpy)

Instantaneous Corrosion Rates Calculated from Equation of Line.
RECOVERY BOILER CORROSION

Carbon Steel Coupons (C1018)

Smelt Present

Equation of line
\[ y = -255 + 235(\ln t) \]
Slope: 4300/t (mpy)

Equation of line
\[ y = -0.8 + 45.4(\ln t) \]
Slope: 825/t (mpy)

Instantaneous Corrosion Rate

Instantaneous Corrosion Rates Calculated from Equation of Line.
Average Values Given in Table
RECOVERY BOILER STUDIES

Carbon Steel Corrosion Coupons (C1018)

No Smelt Present

440°C

Weight Loss (milligrams)

175 mpy

125 mpy

Time (hours)

Front Coupon

Back Coupon

Average Value of Corrosion Rate versus Time
Micrograph of cross section of corrosion scale formed on carbon steel during 24 hour at 440°C. Magnified 500x.
RECOVERY BOILER CORROSION

Stainless Steel Coupons (304)
No Smelt Present

![Graph showing corrosion rate over time.](image)

- **Equation of line**
  \[ y = -17.1 + 11 \ln(t) \]
  - Slope: 200/t (mpy)

- **Equation of line**
  \[ y = 0.9 + 1.3 \ln(t) \]
  - Slope: 23/t (mpy)

*Instantaneous Corrosion Rates Calculated from Equation of Line.*
RECOVERY BOILER CORROSION

Stainless Steel Coupons (304)

Equation of line
\[ y = -29.4 + 27.1 \ln(t) \]
Slope: 485/t (mpy)

Equation of line
\[ y = 5.1 + 1.2 \ln(t) \]
Slope: 21/t (mpy)

Instantaneous Corrosion Rate

Weight Loss (milligrams)

Time (hours)

330°C  440°C

Instantaneous Corrosion Rates Calculated from Equation of Line.
RECOVERY BOILER STUDIES

Stainless Steel Corrosion Coupons (304)

No Smelt Present

Average Value of Corrosion Rate versus Time
RECOVERY BOILER STUDIES
Carbon Steel Corrosion Coupons (440°C)
80% N₂, 20% CO₂

Instantaneous Corrosion Rate

Equation of line
\[ w = 67 + 55 \ln(t) \]
Slope = 1000/t (mpy)

Instantaneous Corrosion Rates Calculated from Equation of Line and Given by Upper Values. Average Corrosion Rate Shown by Straight Line.
Task 3. Continuous Kinetics

Summary of Current Results:

1. CORROSION OBEYS IN THE EARLY STAGE A LOGARITMIC RATE LAW AND AFTER ABOUT ONE DAY BECOMES NEARLY LINEAR (< 500 HR)

2. 10-20% CO₂ RESPONDS TO CORROSION SIMILARLY THAN THE TEST GAS

3. CORROSION SCALE MULTILAYERED WITH TWO DIFFERENTLY STRUCTURED LAYERS

4. CHEMICAL PASSIVATION APPEARED INEFFECTIVE
Task 3., Continuous Kinetics

Future work

1. LONG TERM TESTS WITH THERMOBALANCE
2. TESTS WITH FIXED $P_{O_2}$ AND $P_{S_2}$
Task 4, Local $O_2$ and $S_2$ Activities

Objectives

DEVELOP CAPABILITY TO MEASURE LOCAL $P_{O_2}$ AND $P_{S_2}$ FOR DETERMINATION OF CORROSION CONDITIONS AT METAL SURFACE UNDER SMELT LAYER.
Task 4., Local O₂ and S₂ Activities
Experimental Approach

1. DEFINE LOCAL CONDITIONS
2. CONSTRUCTION OF O₂-PROBE
3. DEVELOP S₂-PROBE
4. CONTINUOUS MEASUREMENTS UNDER SYNTHETIC SMELT
5. SAME FOR FIELD PROBES
Figure 18. Phase stability diagram for system Fe-S-O at 300 and 400 °C

Figure 19. Phase stability diagram for system Cr-S-O at 300 and 400 °C
Task 4., Local $O_2$ and $S_2$ activities

**Progress and Conclusions**

1. $O_2$-PROBE UNDER CONSTRUCTION

2. TEST CONDITIONS CLOSE TO OXIDE/SULFIDE BOUNDARY LINE

3. HIGH TEMPERATURE FAVORS OXIDE OVER SULFIDE