MATERIAL TESTING AND CORROSION EXPERIENCE IN CRUDE UPGRADER ATMOSPHERIC DISTILLATION UNIT

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Presentation Outline

- Introduction
- Background
- Experimental Procedures
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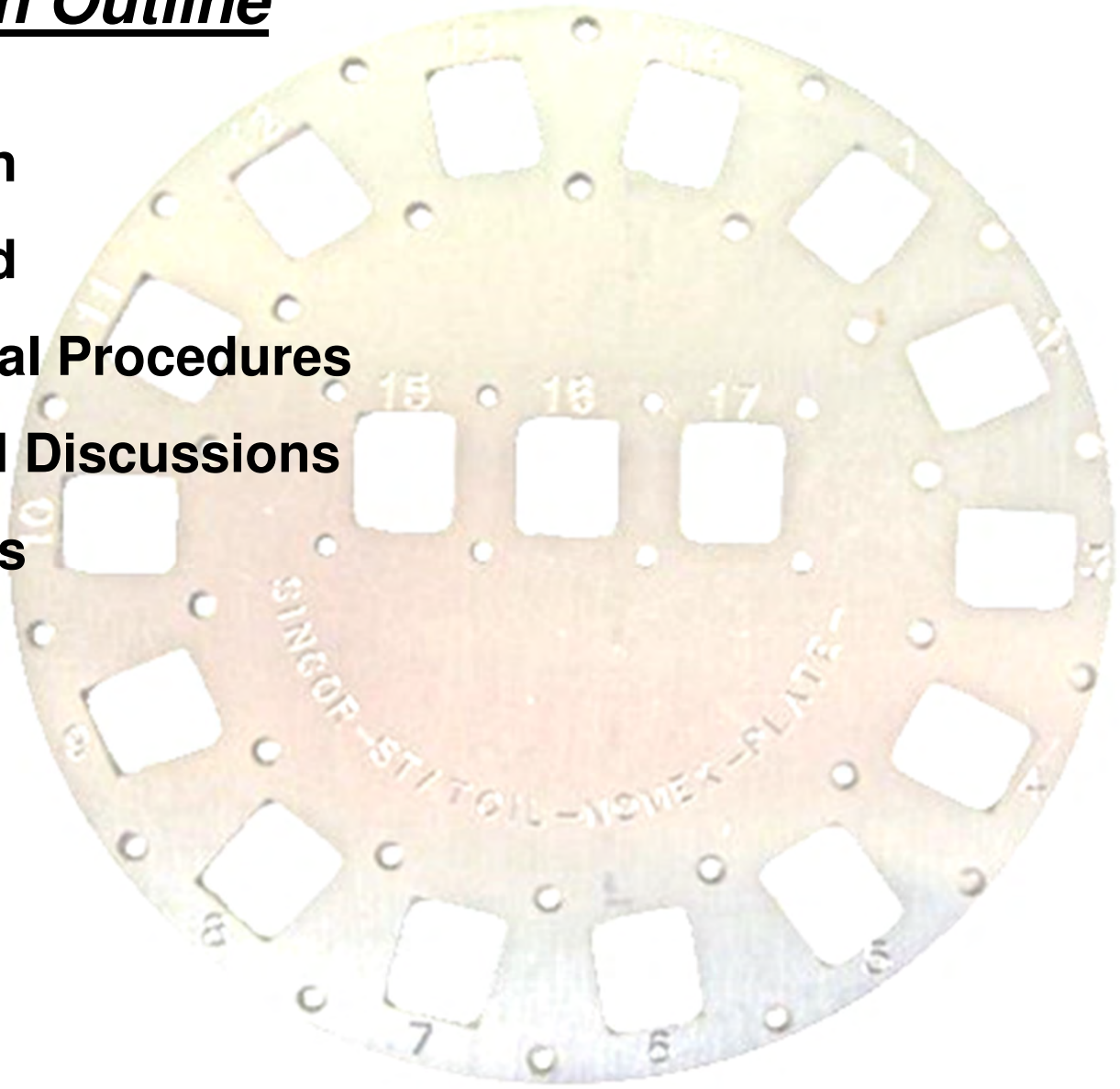
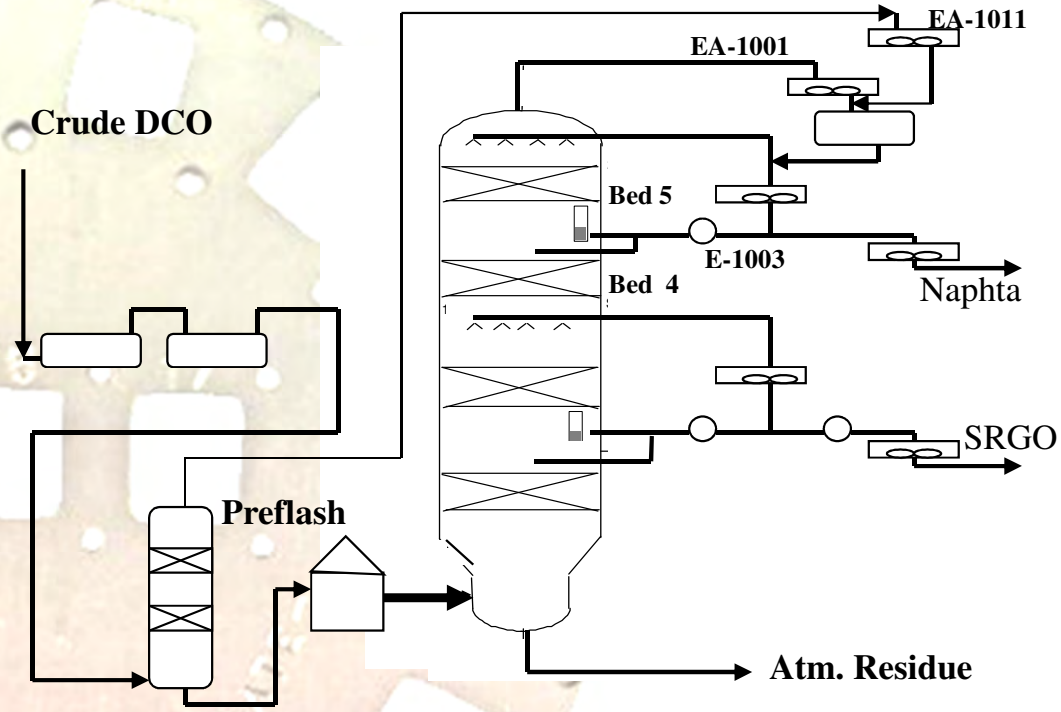


Introduction

Extra heavy oil upgrader plant started operations in 2002.

Two main damage mechanisms were found in the main column: Hydrogen Chloride corrosion, and corrosion due to Wet H_2S and Light Organic Acids in as a consequence of salt deposition



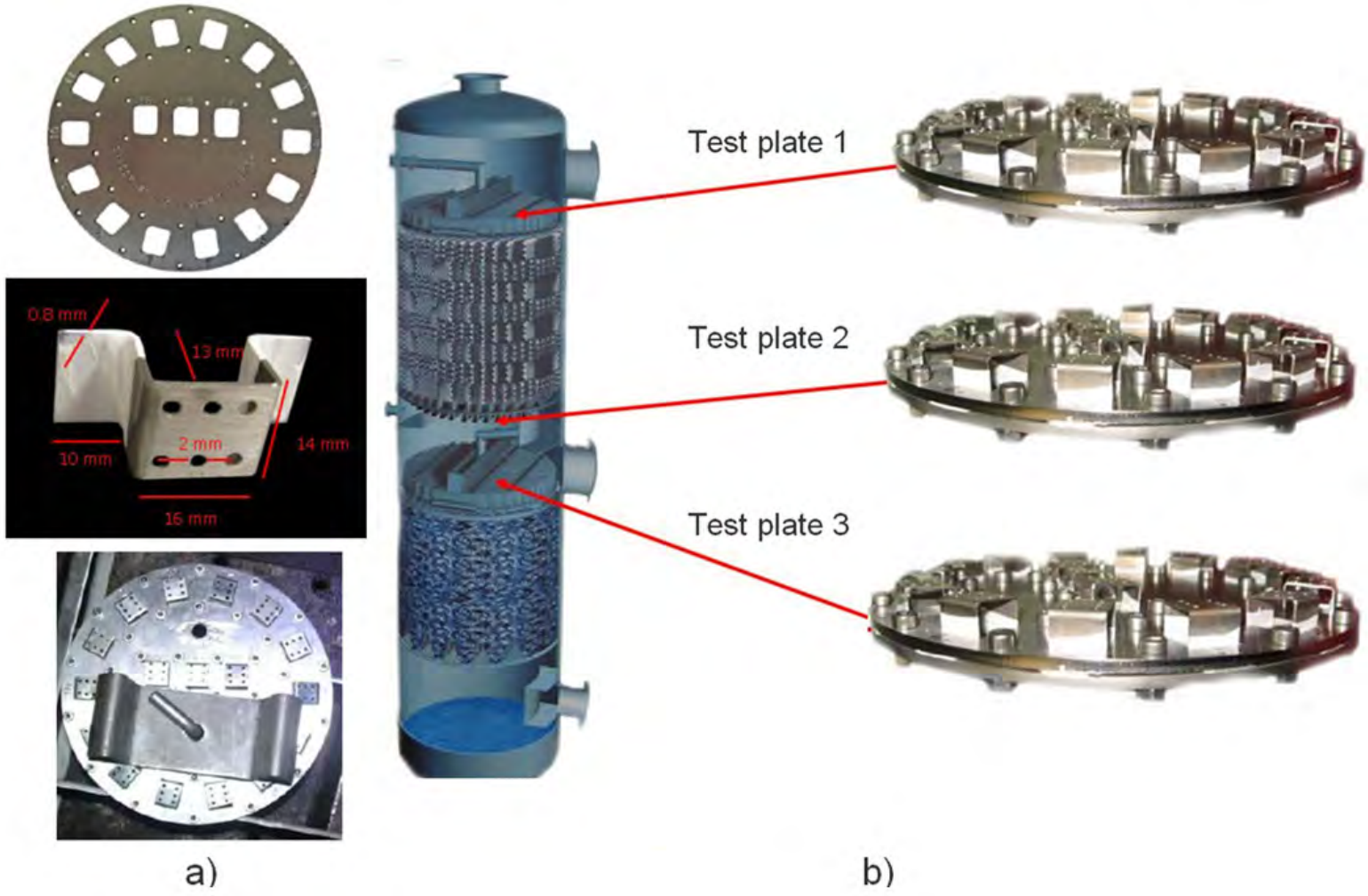
Top packing of the column lost several times after start up.

Tower internals were originally 410 SS. They failed twice.

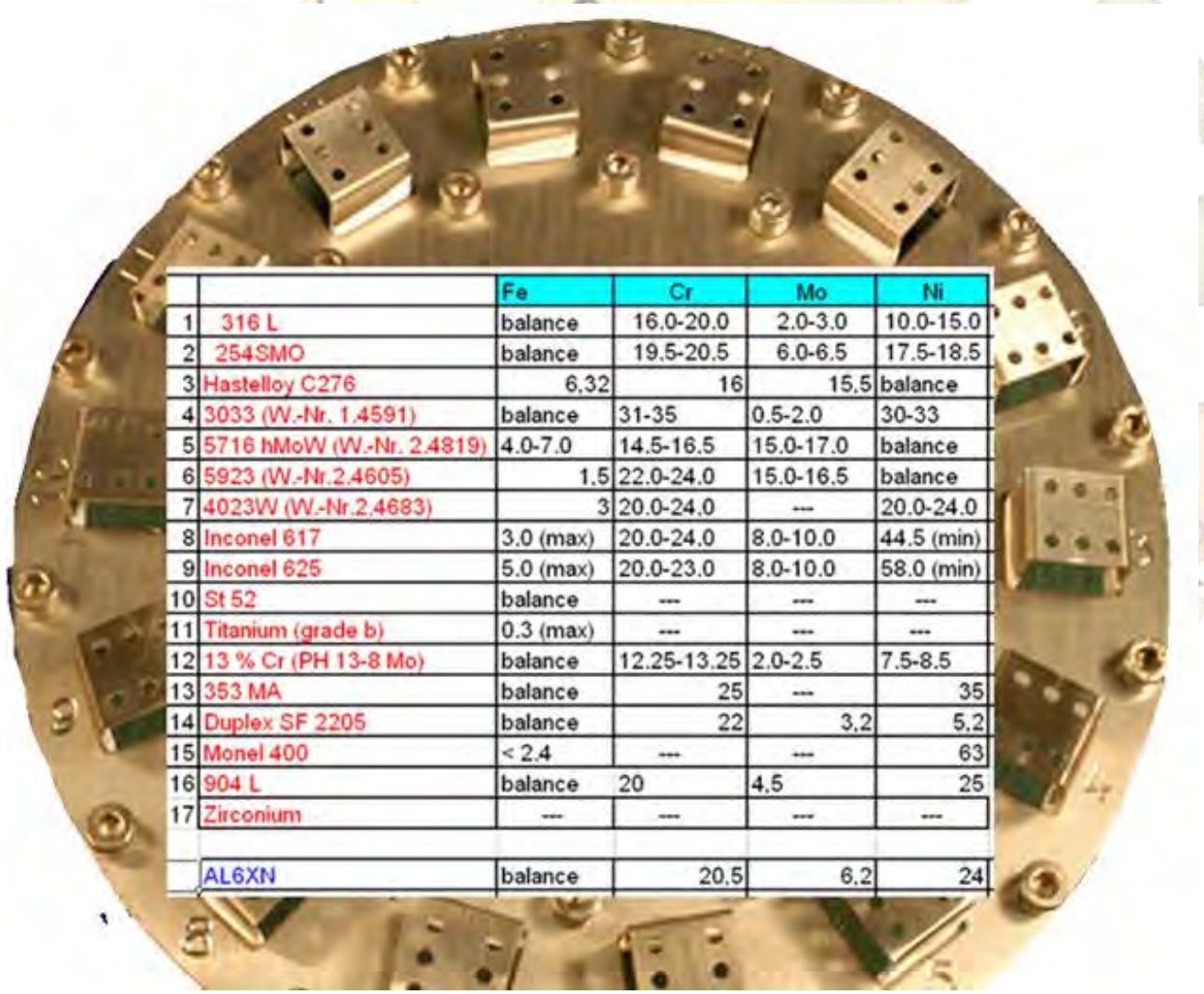
Internals upgraded to AL6XN failed shortly after installation.

Background

Test plates were designed containing 17 different materials. They were installed with the tower internals. The data collected after analysis would be useful for material selection process in crude distillation units of extra heavy crude oils Upgraders.



Most of the coupons were austenitic stainless steel (with a considerable high content of molybdenum) and nickel alloys. Carbon steel, zirconium and titanium were also included.



		Fe	Cr	Mo	Ni
1	316 L	balance	16.0-20.0	2.0-3.0	10.0-15.0
2	254SMO	balance	19.5-20.5	6.0-6.5	17.5-18.5
3	Hastelloy C276	6,32	16	15,5	balance
4	3033 (W.-Nr. 1.4591)	balance	31-35	0.5-2.0	30-33
5	5716 hMoW (W.-Nr. 2.4819)	4.0-7.0	14.5-16.5	15.0-17.0	balance
6	5923 (W.-Nr.2.4605)	1.5	22.0-24.0	15.0-16.5	balance
7	4023W (W.-Nr.2.4683)	3	20.0-24.0	---	20.0-24.0
8	Inconel 617	3.0 (max)	20.0-24.0	8.0-10.0	44.5 (min)
9	Inconel 625	5.0 (max)	20.0-23.0	8.0-10.0	58.0 (min)
10	St 52	balance	---	---	---
11	Titanium (grade b)	0.3 (max)	---	---	---
12	13 % Cr (PH 13-8 Mo)	balance	12.25-13.25	2.0-2.5	7.5-8.5
13	353 MA	balance	25	---	35
14	Duplex SF 2205	balance	22	3,2	5,2
15	Monel 400	< 2,4	---	---	63
16	904 L	balance	20	4,5	25
17	Zirconium	---	---	---	---
	AL6XN	balance	20,5	6,2	24

The test plates were exposed for 1.3 years (approximately 11400 hours). Main laboratory analyses were made at PDVSA Intevep.

Experimental Procedures

After taking out the test plates, cleaning and visual inspection were done. T**he probes were divided in two equal parts; one for measuring corrosion rate and the other half for microstructure analysis.**

Each probe with its corrosion products was weighted. Corrosion layer was removed as per ASTM G1 and each probe was weighted again without the corrosion layer.

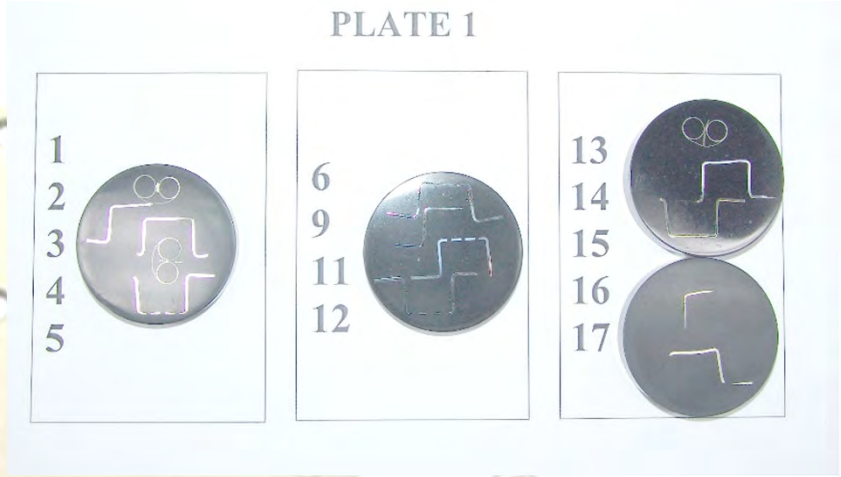
G1-03

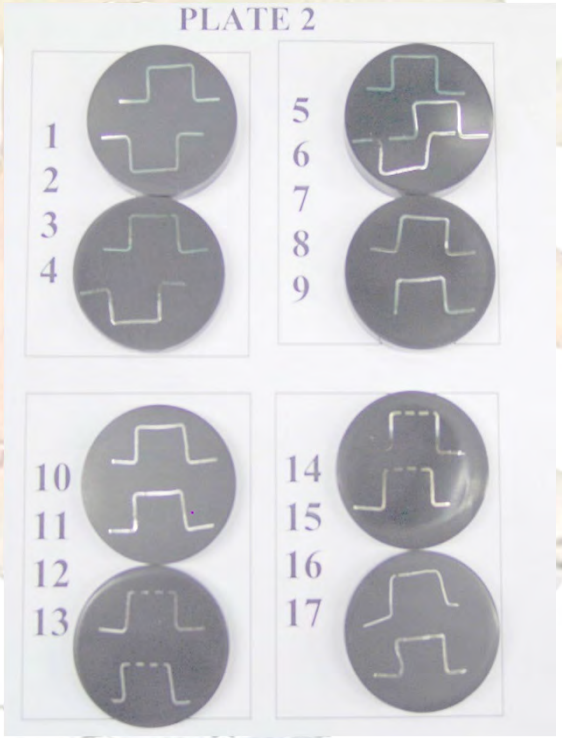
Experimental Procedures

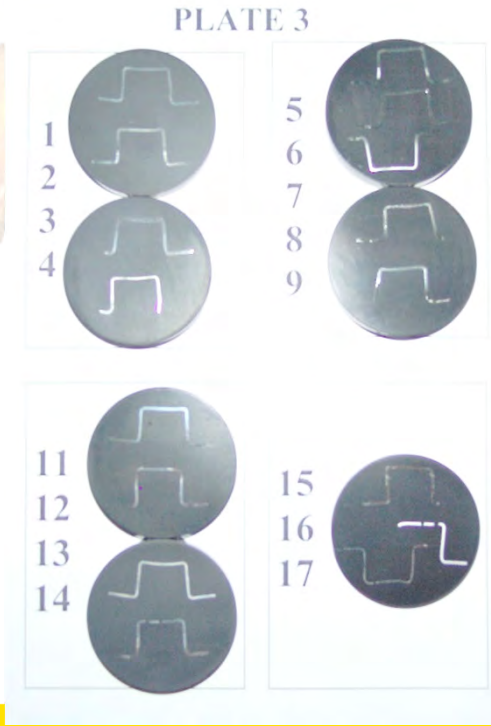
Metallographic samples were prepared for microstructure analysis.

Scanning Electron Microscopy (SEM) was used to find out the corrosion product elements and their distribution by means of electron image by means of Energy Dispersive Spectrum (EDS) and X ray dot maps.

Optical microscope (OM) was for evaluating grain size and microstructure morphology.



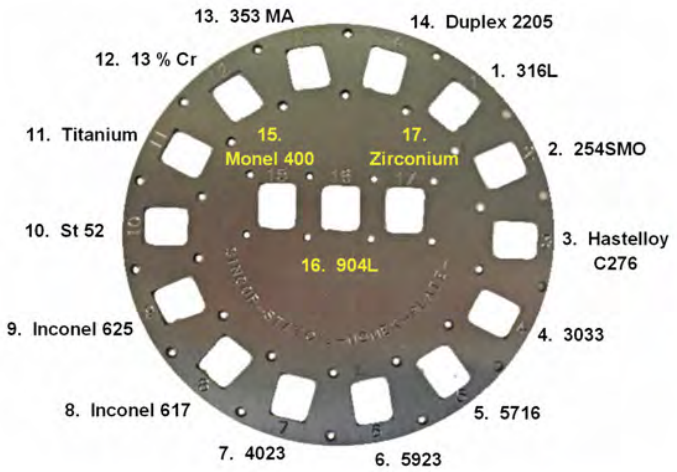




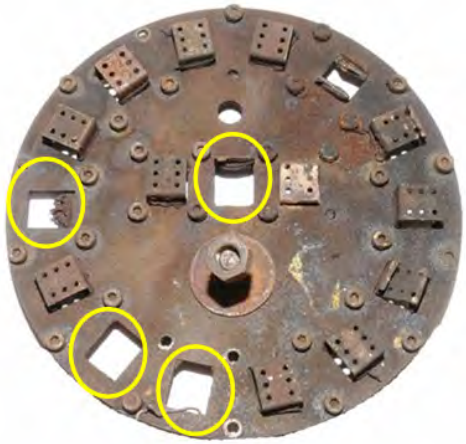
Results and Discussions

Visual Inspection

Test plate 1, several coupons were lost (4023W, Inconel 617, Carbon Steel and 904L) perhaps due to the corrosion conditions prevailing nearby the overhead section of the column. Test plate 3,only carbon steel coupon was lost.



a)



b)

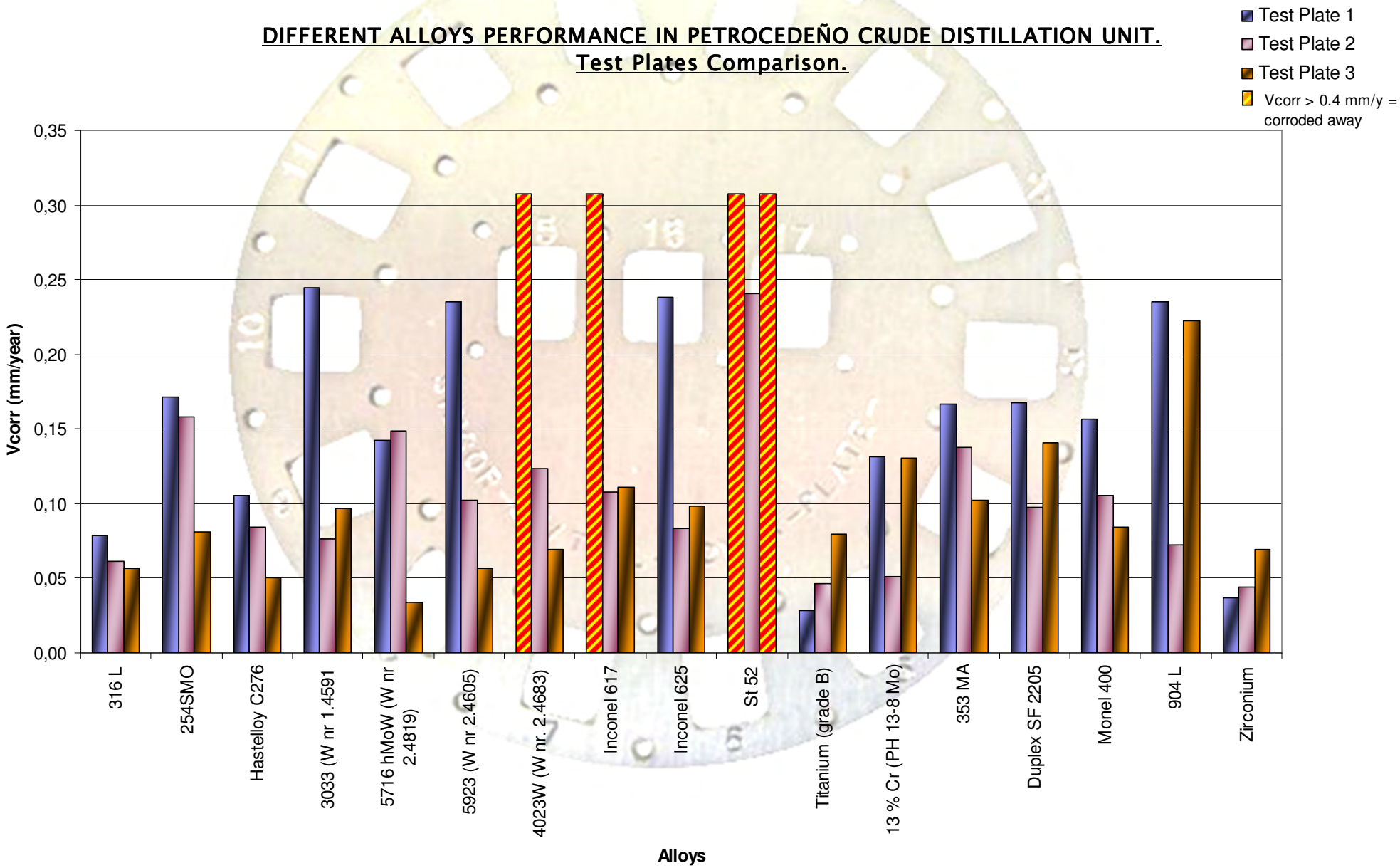
c)

d)

a) Distribution of the coupons or probes before being exposed to operation conditions. b) Test plate 1 after being exposed showing 4 missing coupons. c) Test plate 2 after exposition. No missing probes. d) Test plate 3 after being exposed showing 1 probe lost.

Results and Discussions

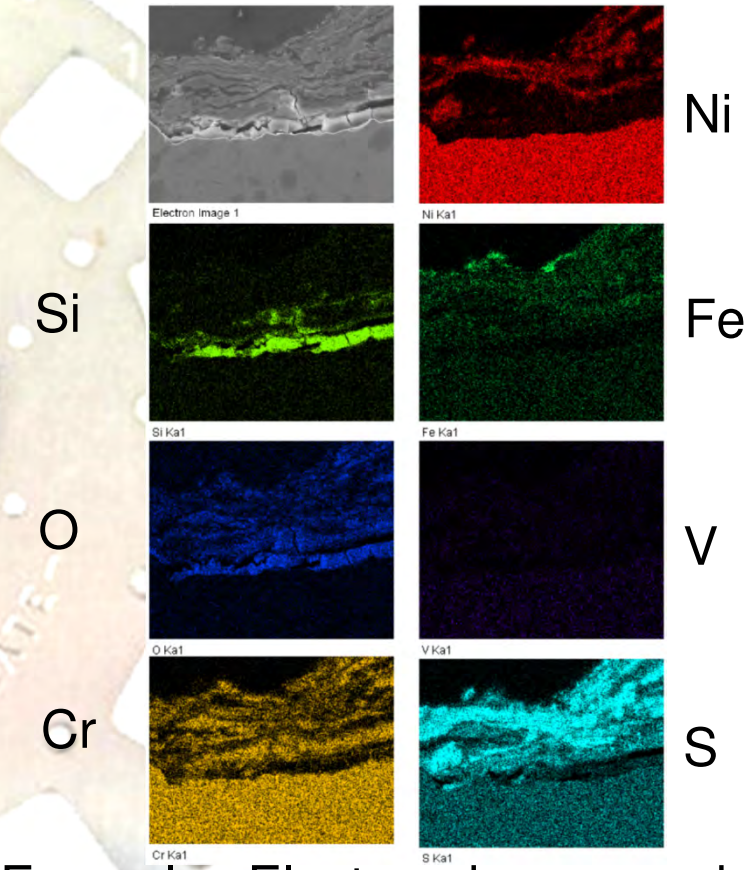
Corrosion rate determination



Results and Discussions

Scanning Electron Microscopy (SEM)

ALEACIÓN	ELEMENTOS PRESENTES EN LA CAPA DE PRODUCTOS		
	Dispositivo 1	Dispositivo 2	Dispositivo 3
316 L	Cr, S, Ni, O, Cl	O, S, Fe, Cr, Ni	Fe, Ni, S, Cr, Mo, O
254 SMO	O, S, Cr, Ni, Cl	O, S, Cr, Fe, Ni	Fe, S, O, Mo
Hastelloy C276	O, S, Cr, Fe, Ni, W, Co	Fe, S, Cr, O	S, Fe, Cr, O, Mo
3033	S, Cr, Ni, O, Cl	O, S, Fe	Fe, Cr, S, O, Mo
5716 hMoW	Fe, O, Si, S, Mo	Fe, O, Si, S, Mo	Fe, O, W, Cr
5923	Ni, Cr, Si, Fe, S, O, V	Si, Fe, S, O, Mo	Si, Fe, O, Mo
4023W	Sin probeta	S, Cr, O, Co, Fe	S, Cr, O, Fe, Co, W
Inconel 617	Sin probeta	S, Cr, O, Fe, Co, Mo	S, Cr, Ni, O, Fe, Co, Mo
Inconel 625	Ni, Cr, S, Si, O, Fe	S, Fe, Si, O, Mo, Cr, Si	Ni, S, Fe, O, Mo
St 52	Sin probeta	Si, Fe, S, O, Cl	Sin probeta
Titanio Gr B	Ti, O, S, Na, Ca, Si	Fe, S, O, Cr	S, Fe, O
13% Cr Mo	Ni, Fe,Cr, Si, S, O, Na, V, Ca	Fe, Si, S, Ni, O, Cr, Co	Si, Fe, S, Cr, O, Ni, Mo
353 MA	S, Ni, Fe, Si, Cr. O	Ni, Si, S, Fe, Cr, O	Fe, O, Mo, S, Cr, Ni
Duplex SF 2205	Si, Ni, S, Cr, O, Na	S, Fe, Cr, O, Ni	Fe, Cr, Ni, O, S, Mo
Monel 400	S, Ni, O, Cu, Fe, Cr	S, Ni, O, Cu, Fe, Cr	S, Si, Ni, Fe, O, Cu, Cr
904 L	Ni, S, Cr, Si, O, Cu	S, Fe, Si, O, Mo	Fe, Ni, S, Cr, O, Mo
Zirconio	Fe, Zr, O	Fe, O, S, Cr	Fe, O, S



Example: Electron image and X ray dot maps of the corrosion product formed on 5923 alloy located in test plate 1

PDVSA Statoil

Results and Discussions

Microstructure characterization with Optical Microscopy (OM)

Alloy	Cracking	Thickness loss	General Observations
316 L	✓		Massive cracking in coupons located in test plate 2
254 SMO	✓	✓	High carbide, nitro carbide and manganese presence
Hastelloy C276			Low thickness loss. Estimated remaining life was over 5 years even for test plate 1 (more aggressive conditions)
3033		✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions).
5716 hMoW			Very low thickness lost because estimation times were over 5 years in all probes
5923		✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions).
4023W		✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions). It was found sigma phase in high percentage.
Inconel 617		✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions).
Inconel 625		✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions).
St 52		✓	High thickness loss in all test plates. Estimated remaining life was below 5 years.
Titanium Grade B			Possible titanium hydride formation
13% Cr Mo	✓		Pitting
353 MA	✓	✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions)
Duplex SF 2205	✓	✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 (more aggressive conditions)
Monel 400			Some irrelevant pits
904 L	✓	✓	High thickness loss. Estimated remaining life was below 5 years for coupon located in test plate 1 and 3.
Zirconium			Exceptional performance

Conclusions

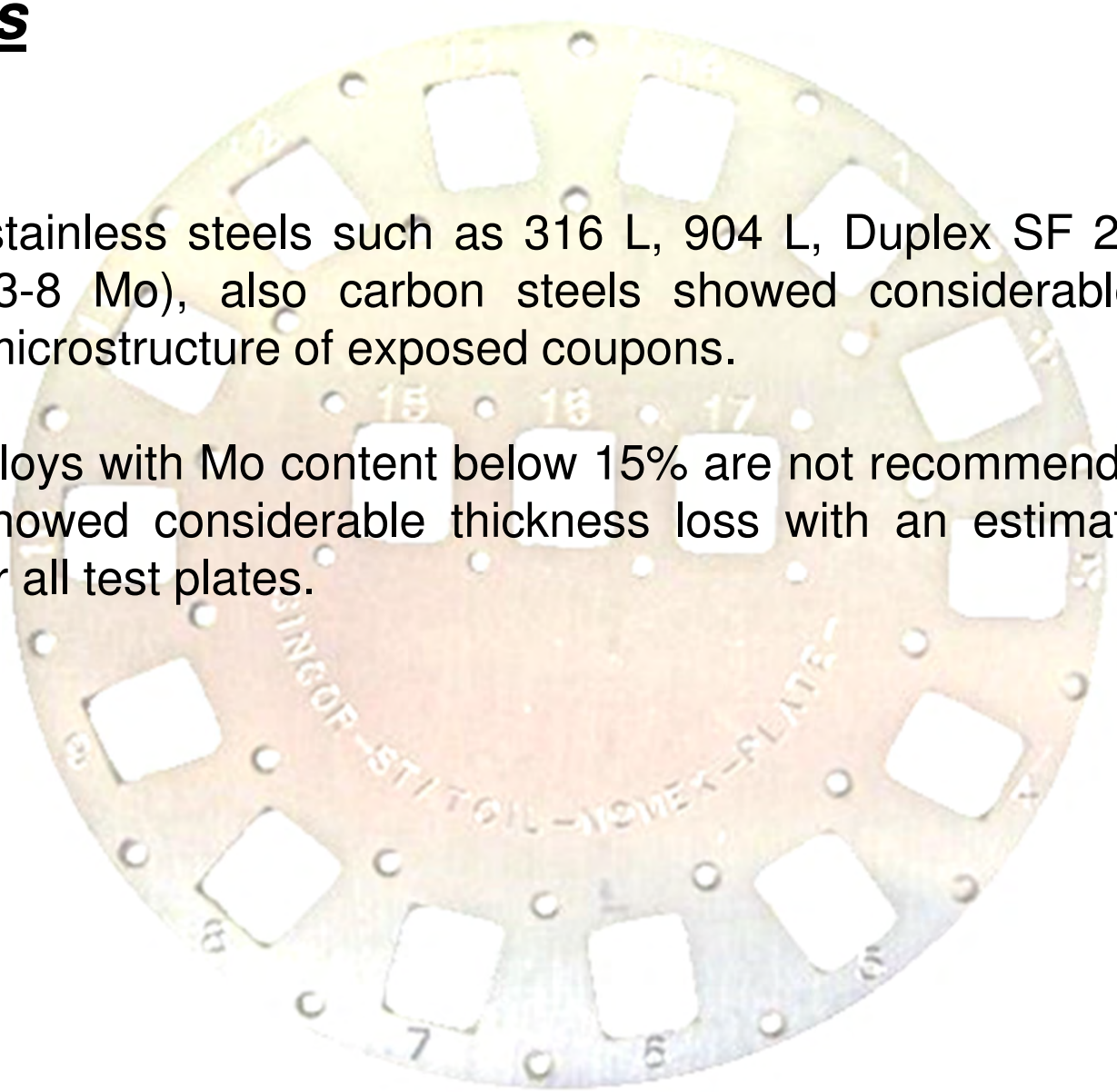
- The material selection for the operating conditions of this atmospheric distillation column is: Hastelloy C276, stainless steel super austenitic 5716 hMoW, Titanium Grade B, Monel 400 and Zirconium.

- Hastelloy C276, Titanium Grade B and Zirconium showed the best performance from corrosion rate stand point.

- Coupon corrosion product layers showed presence of oxygen and sulfur over chloride, calcium and sodium salts, suggesting wet H_2S as common mechanism. The presence of Mo in the corrosion product layer of the 5923 alloy could be a promoter of a more protective one.

- Austenitic stainless steel and nickel super alloys with high molybdenum content (over 15 %) showed good performance.

Conclusions



- Conventional stainless steels such as 316 L, 904 L, Duplex SF 2205, 353 MA and 13% Cr (PH 13-8 Mo), also carbon steels showed considerable thickness loss. Cracking in the microstructure of exposed coupons.

- Nickel super alloys with Mo content below 15% are not recommended for this service because they showed considerable thickness loss with an estimated remaining life below 5 years for all test plates.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