CORROSION BEHAVIOR AND/OR DEGRADATION OF MATERIALS USED IN THE TRANSPORT OF SUPERCRITICAL CO_2 ($SCCO_2$), IN CONTACT WITH AQUEOUS SOLUTION WITH CO_2 WITH/WITHOUT THE PRESENCE OF CONTAMINANTS

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Research Centre
for Gas Innovation
cleaner energy for a sustainable future

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What is project 40 about?

- HSLA steels are one of the most used materials for the production of pipes in the oil and gas industry.
- It can be found a complete set of materials selection standards for oil and gas transportation. Nevertheless, those standards can not be applied directly for $SCCO_2$ (pressure increases from 2.0 MPa to 8.0 MPa).
- There is not a complete set of standard procedures to select materials for Carbon Capture and Storage (CCS).
- The literature survey shows that there is an increasing interest on the investigation of the corrosion behavior of steel in SCCO2. Some important issues: to trigger the corrosion process water condensation is necessary;
  - Data are conflicting about the maximum amount of water that is necessary for condensation to take place;
  - Acidic contaminants in the $SCCO_2$ stream contribute to accelerate the corrosion rates. However, information on the maximum quantities of contaminants that can be transported safely are conflicting.

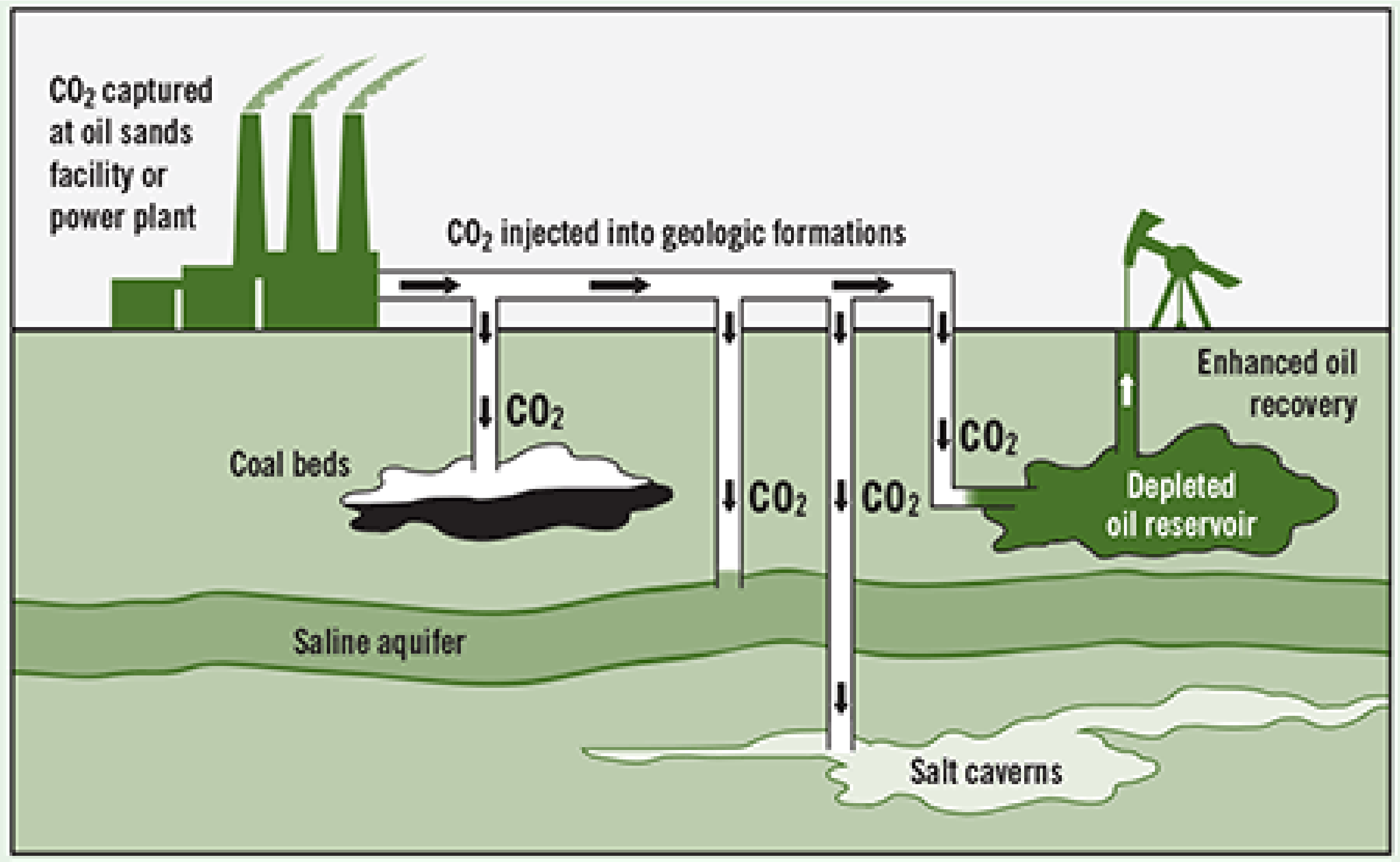
What is project 40 about?

- The Project will focus both in the evaluation of the electrochemical properties (corrosion behavior) of HSLA steels and in composites degradation to be used in $SCCO_2$ transportation and also in the determination of the maximum concentrations of contaminants that can be transported safely (with no increase in the corrosion /degradation rate)

- This project also will compile the existing literature about corrosion and degradation of materials used in $SCCO_2$ transportation as well as those in development.

- The behavior of trading and developing HSLA steels and composite materials will be studied in the Project 40.

Our focus is the pipeline used for transportation!



Source: www.oag-bvg.gc.ca/internet/Francais/parl_cesd_201212_04_f_37713.html

METODOLOGY

Carbon steels

Steel	C	Mn	Si	S	P	Ni	Cu	Cr	Al	Nb	V + Ti
X65E	0.04	1.37	0.35	0.001	0.009	0.06	0.05	0.03	0.04	0.05	0.02
X65LMn	0.038	0.39	0.139	0.001	0.009	0.137	0.28	0.421	0.037	0.085	0.0143
A36	0.25	1.20	0.40	0.050	0.004	--	0.20	--	--	--	---

I and II. Inclusions analisis and microestrutural characterization

Preparation, analysis and classification of inclusions according to ASTM E45- 13

Samples cut parallel to the lamination direction - sanded to # 1200 and polished to 1μm.

Analysis by MO and MEV- Chemical composition estimated by EDS.

Microstructural characterization according to ASTM E112-13

Samples cut perpendicular to the rolling direction - sanded up to # 1200 and polished up to 1μm.

Attack with Nital 2% - analysis by MO and MEV, Chemical composition estimated by EDS.

III. Electrochemical tests (EIS)

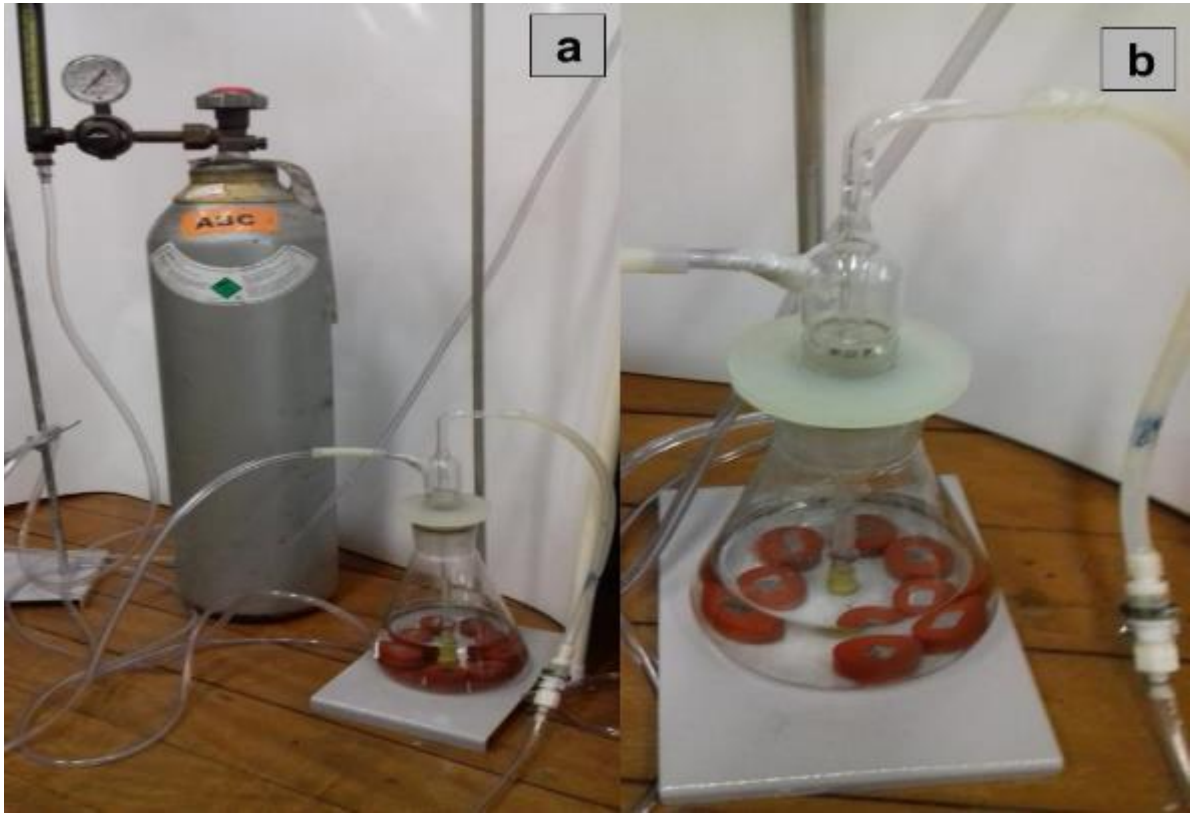
Evaluation of Impedance Measures Low ASTM E112-13

Three electrode system: ER (ECS); CE (graphite bar); WE (sample buffed in bakelite, specimens cut by cross section to lamination direction), Exposed area 1 cm2.

All electrochemical tests were performed in deionized water and saturated with CO2, the initial pH was 4.69 ± 0.01. There was a minimum difference in the pH value throughout the test.

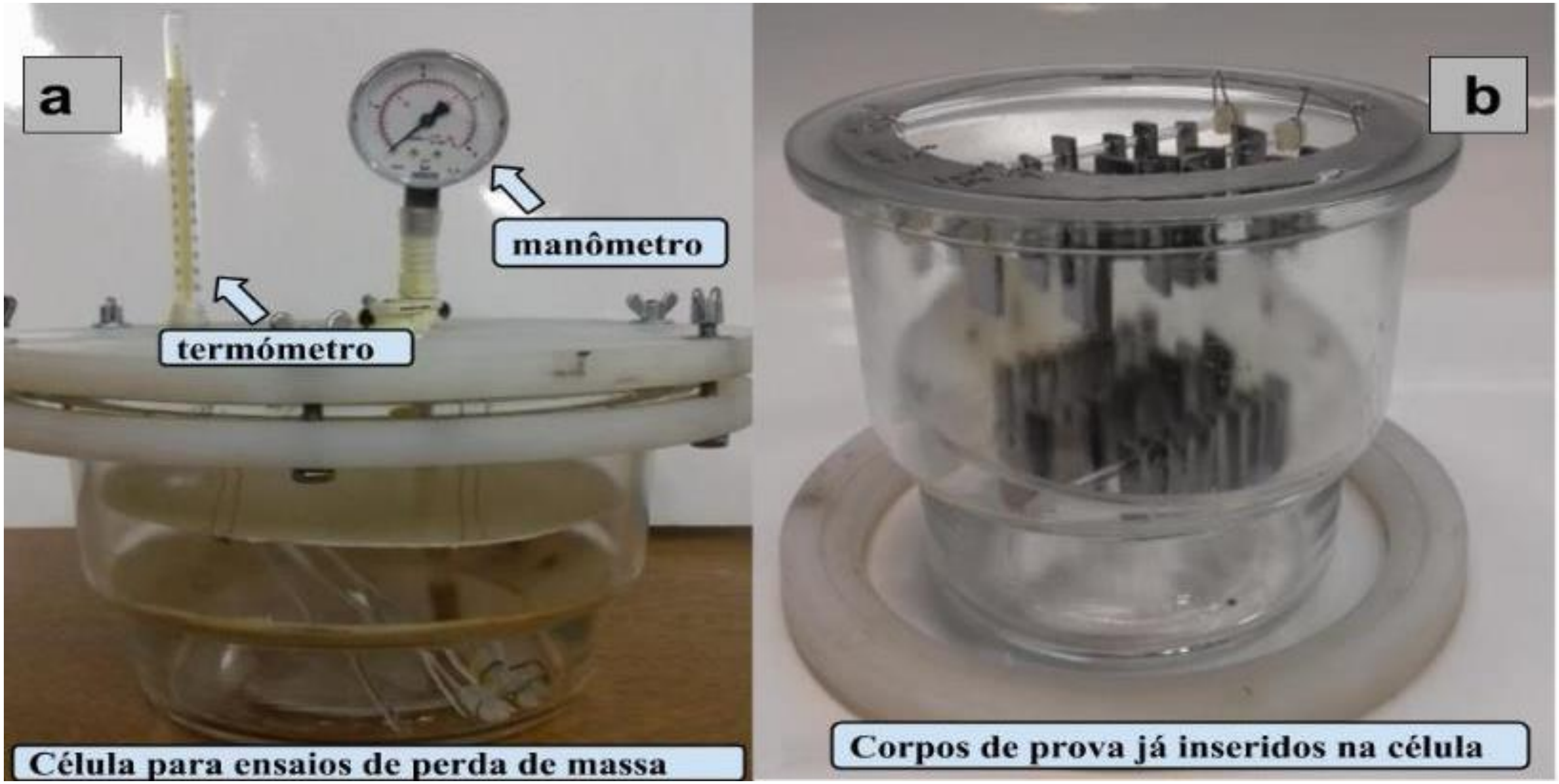
IV. Corrosion morphology (Oxide scale)

In order to evaluate in greater depth the evolution of corrosion in the three materials, SEM tests of the morphology of the corrosion products of the steels after immersion for 3 and 5 days in a solution of distilled and deionized water saturated with CO_2 were performed.

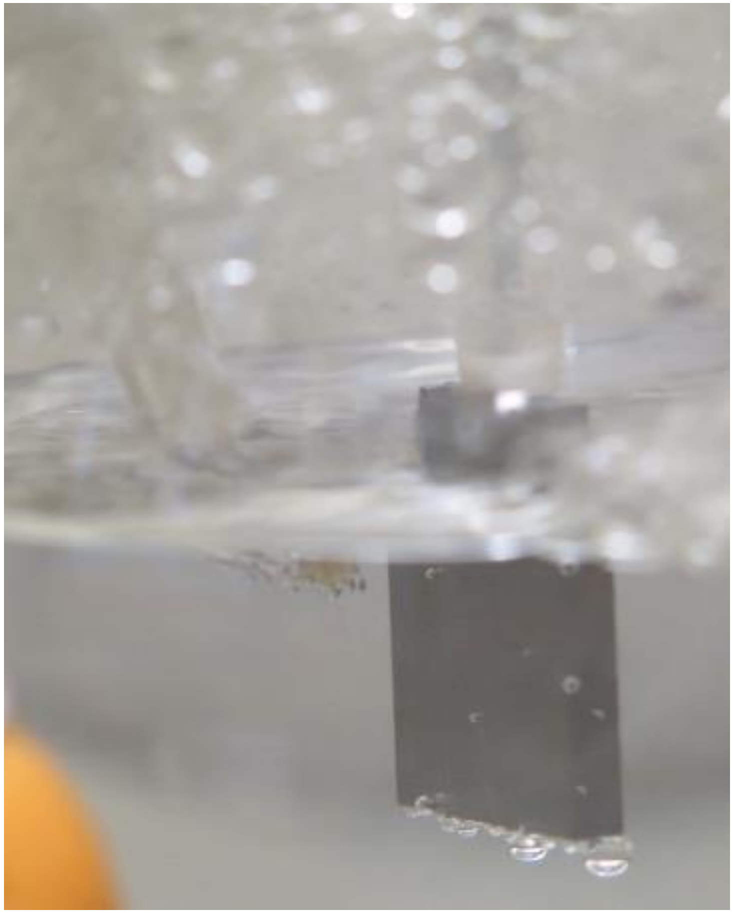


V. Weight loss tests

Weight loss tests were carried out in order to calculate the corrosion rate (m / year) by means of this technique and to compare with the results obtained by techniques electrochemistry.



Electrochemical tests: EIS/Potentiodynamic polarization



RESULTS

I. METALLOGRAPHY AND INCLUSION CHARACTERIZATION

1. Analisis of X65E steel inclusions

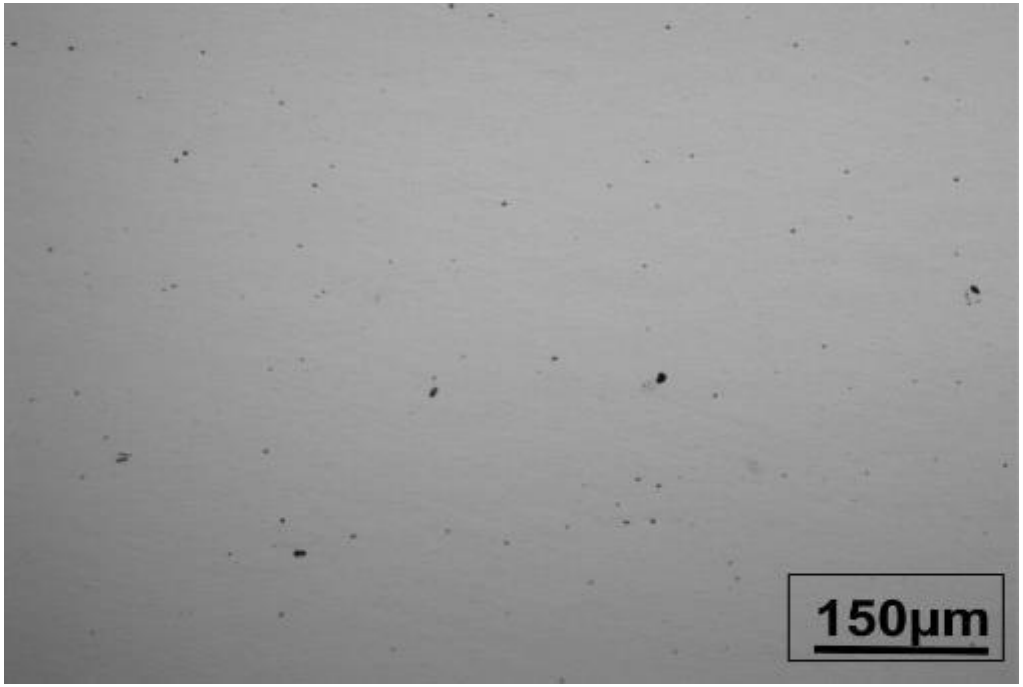
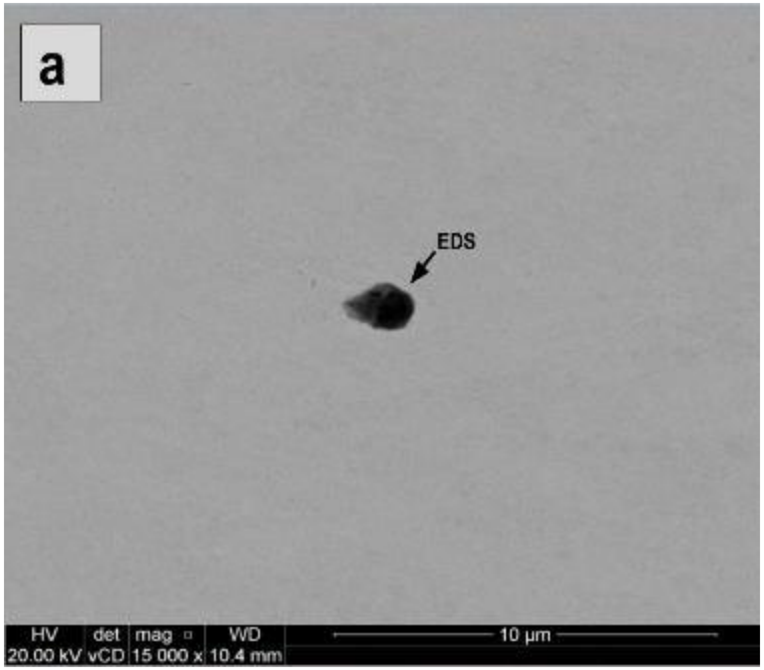


Figure - OM Image OF distribution of inclusions in steel API 5L X65E.
Polishing 1µm without attack.

EDS Analisis of X65E steel inclusions

The average size is between 1.8 and 5.8 μm. Classification as D-globular oxide-sulfides, fine series



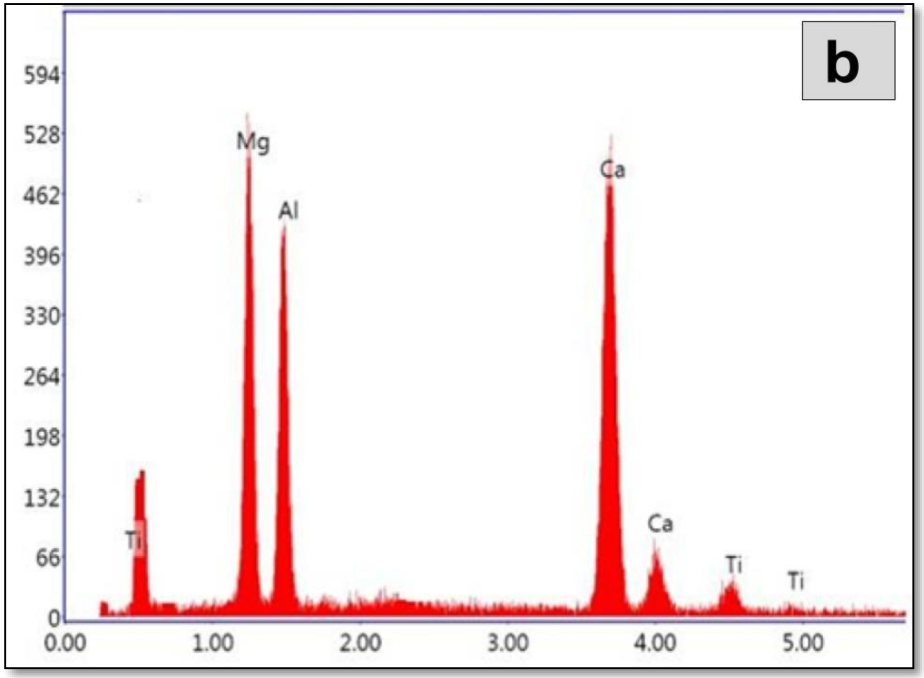


Figure – (a) Electron backscattered image of a X65E steel inclusion. (b) Chemical analysis by ESD of the inclusion indicating the presence of Ca, Al and Mg.

2. Analisis of X65LMn steel inclusions

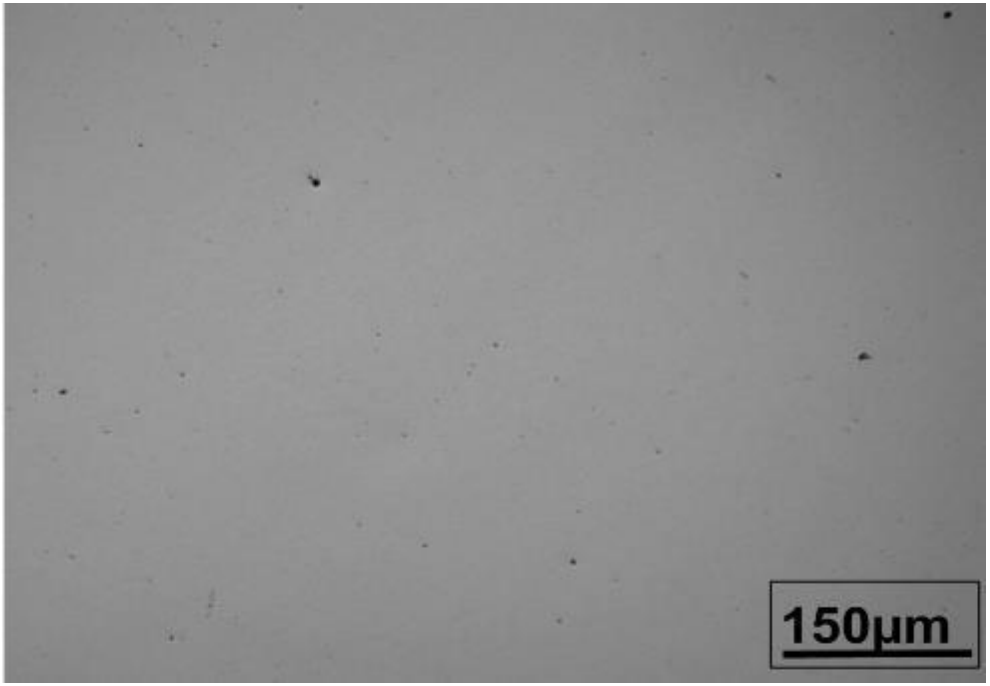


Figure - Image by MO with the distribution of the inclusions in steel API 5L X65LMn
Polishing 1µm without attack. Longitudinal section to the rolling direction.

EDS analisis of 65LMn steel inclusions

The inclusions average size is between 1.3 and 5.6 μm. Classification as D-globular oxide-sulfides, fine series.

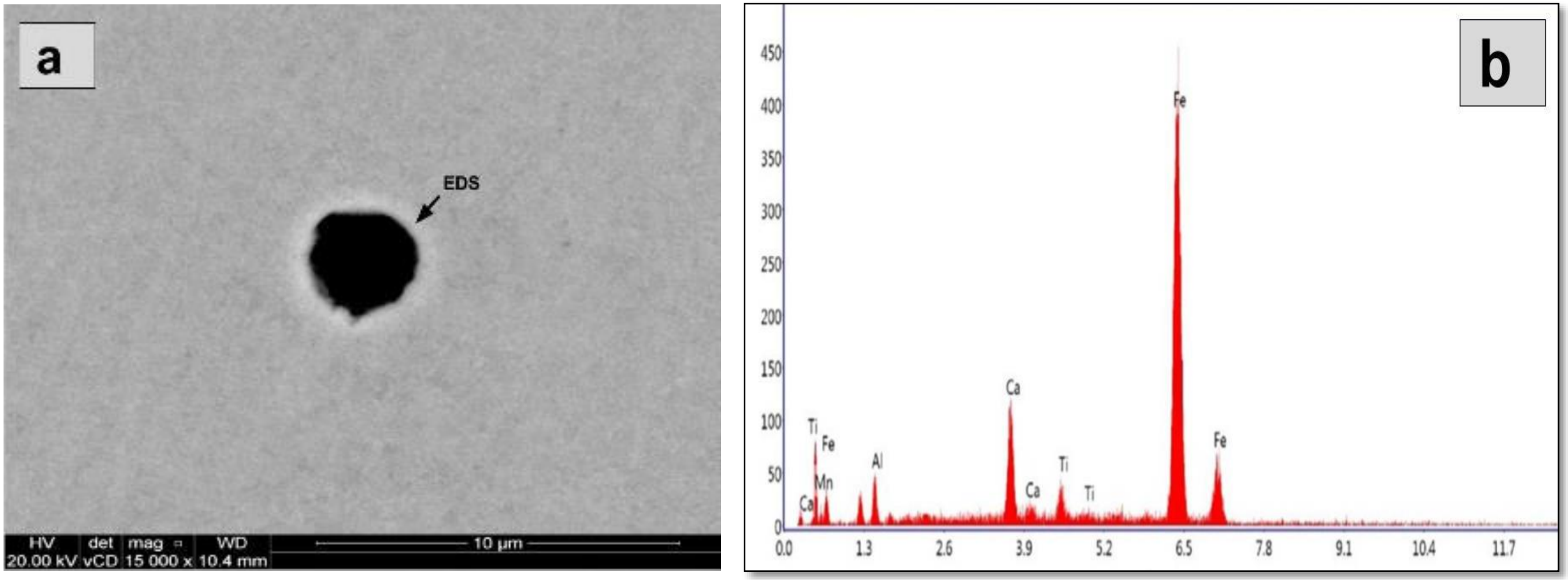


Figure - (a) Image of backscattered inclusion electrons found in steel X65LMn. (B) Chemical analysis by ESD of the region showing to be inclusion of Ca, Al, Ti and Mn.

3. Inclusion analisis of A-36 Steel inclusions

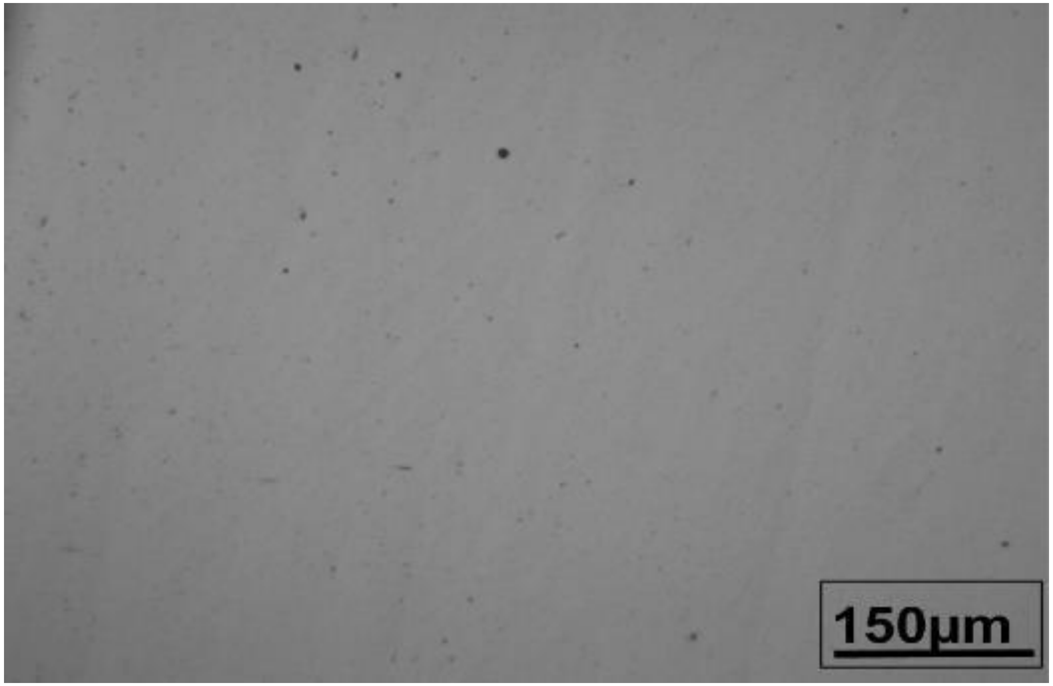


Figure - OM image with the inclusions distribution in ASTM A-36 steel. Polishing 1µm without attack. Longitudinal section to the rolling direction.

ESD analisis of A-36 Steel

The average size is between 1.5 and 5, 4 μm. Classification as D-globular oxide-sulfides, fine series.

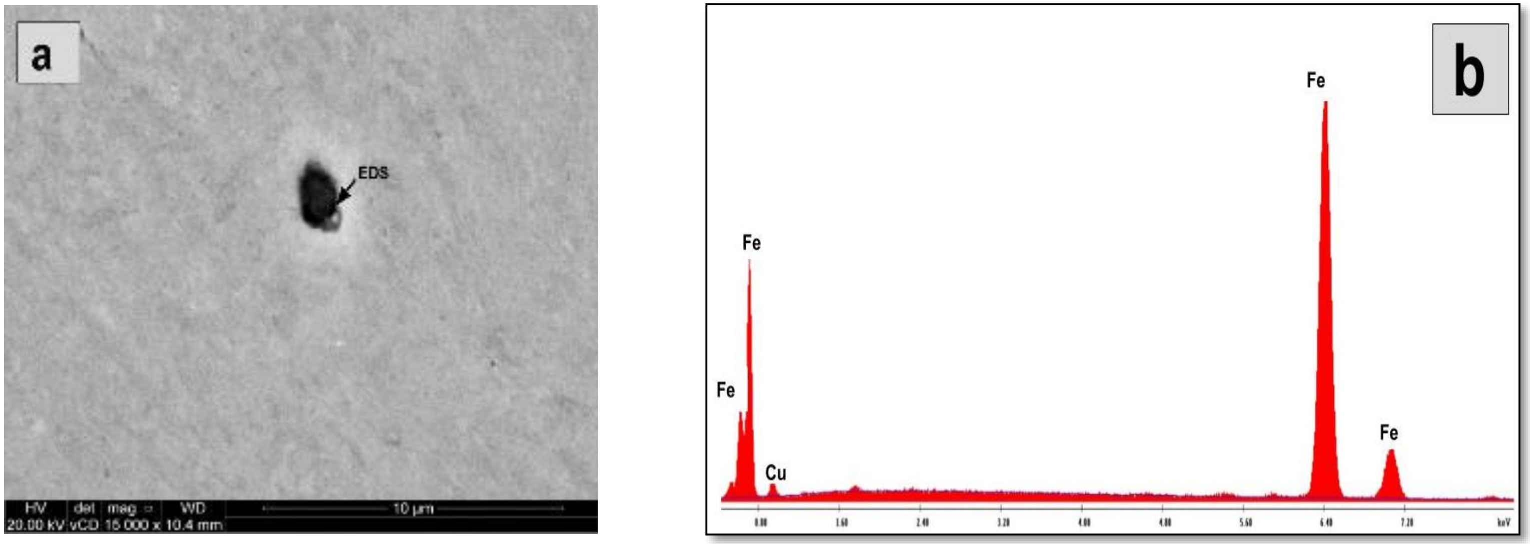


Figure - (a) Image of backscattered electrons of inclusion found in ASTM A36.
(b) Chemical analysis by ESD of the region showing that it is inclusion with presence of Cu.

II. RESULTS: METALOGRAPHY

1. Microstrcutural characterization: API 5L X65E Steel

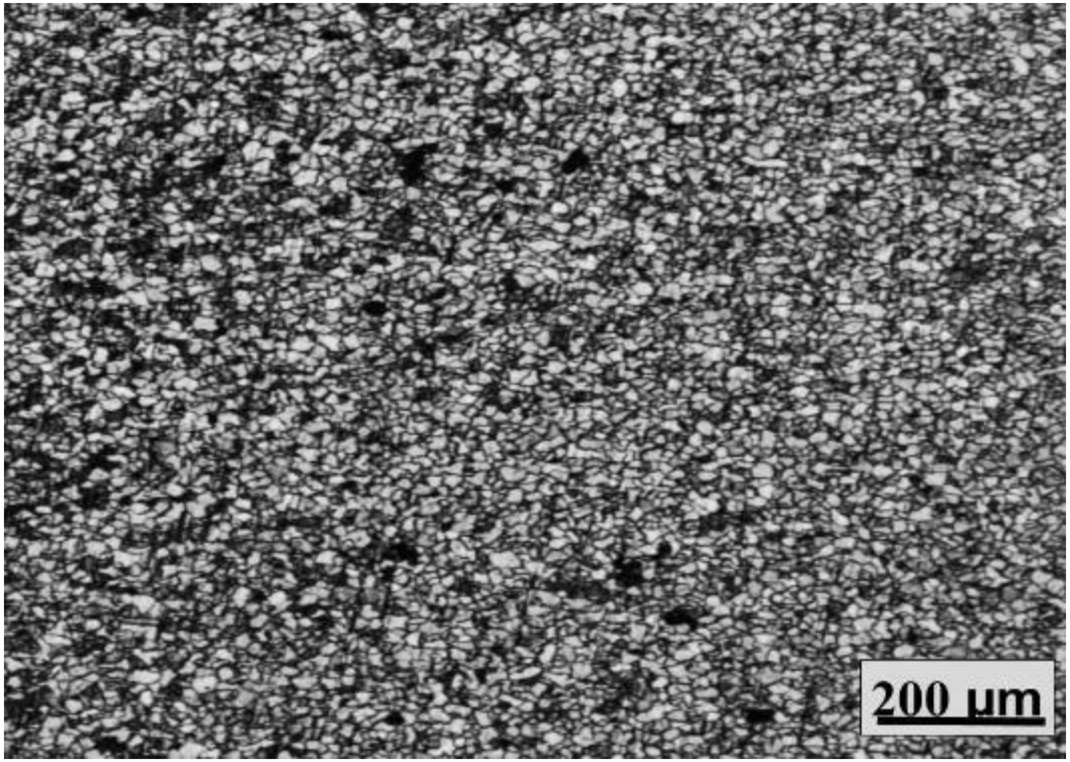
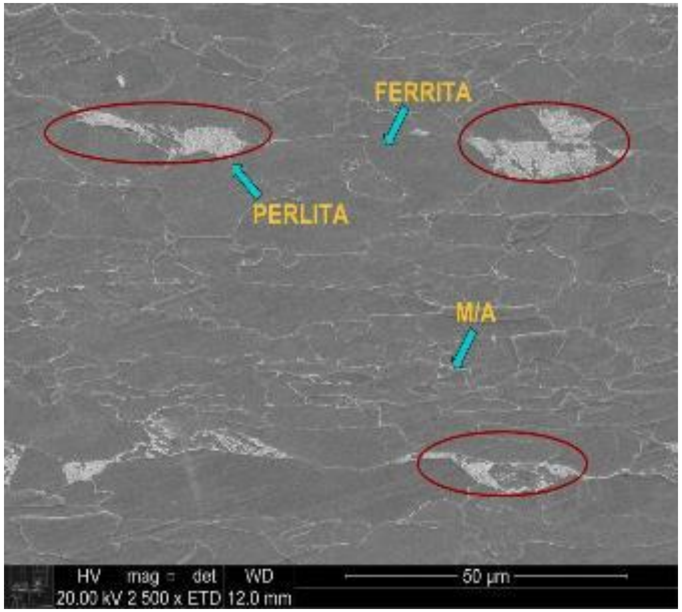
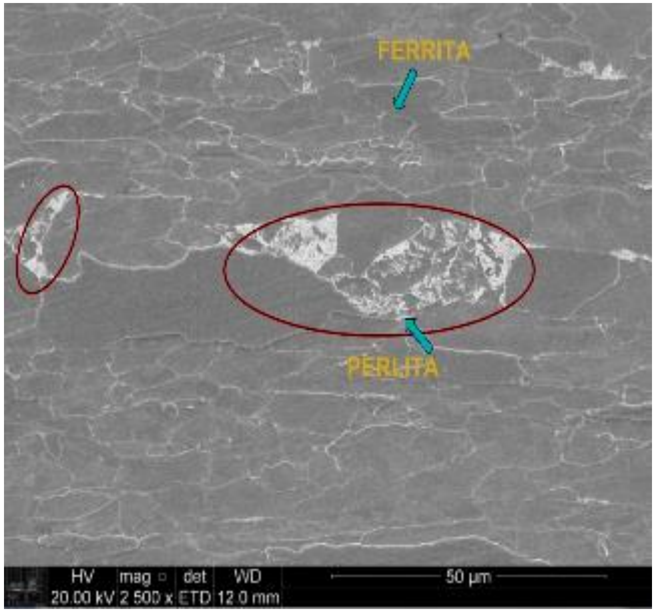


Figure - Optical microscopy of the central region of the cross section of API 5L X65E steel. Attack: Nital 2%.

Microestrutural characterizaton: API 5L X65E Steel



(a)



(b)

Figure - Optical microscopy of the central region of the cross-section (a) and longitudinal (b) of API 5L X65E steel. Attack: Nital 2%. Grain size of medium 6.5 ± 0.3 µm, using circular intercepts method (ASTM INTERNATIONAL E112-13, 2014) with the help of the ImageJ program.

2. Microestrutural characterization: API 5L X65 LMn steel

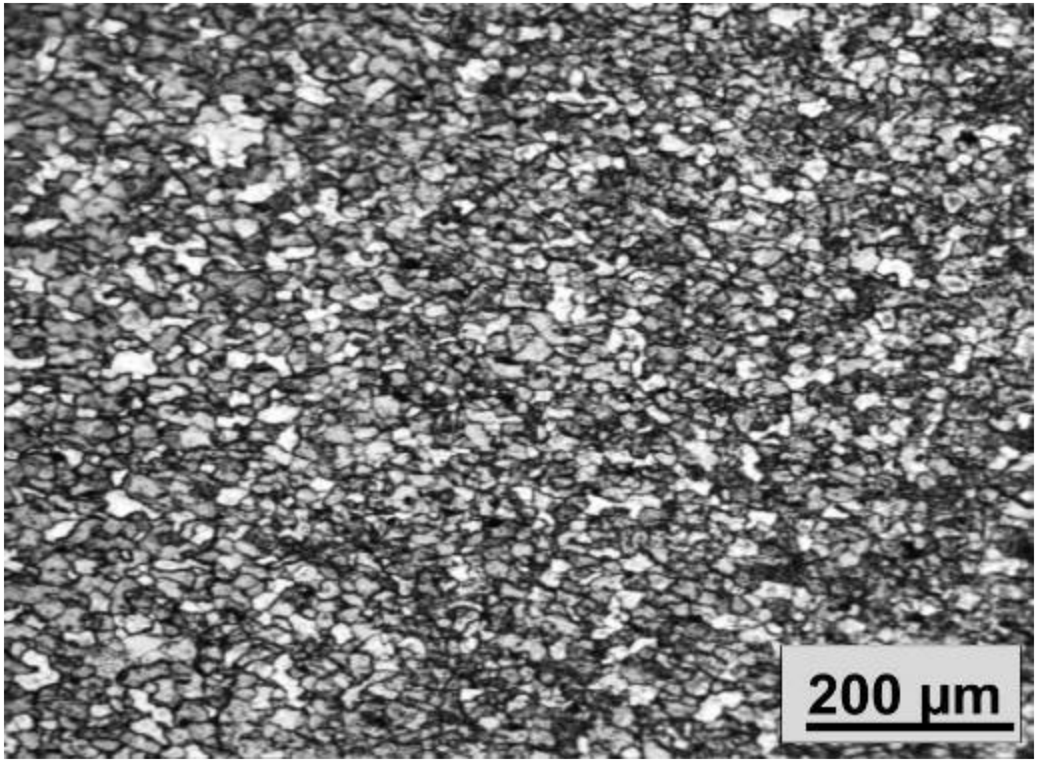
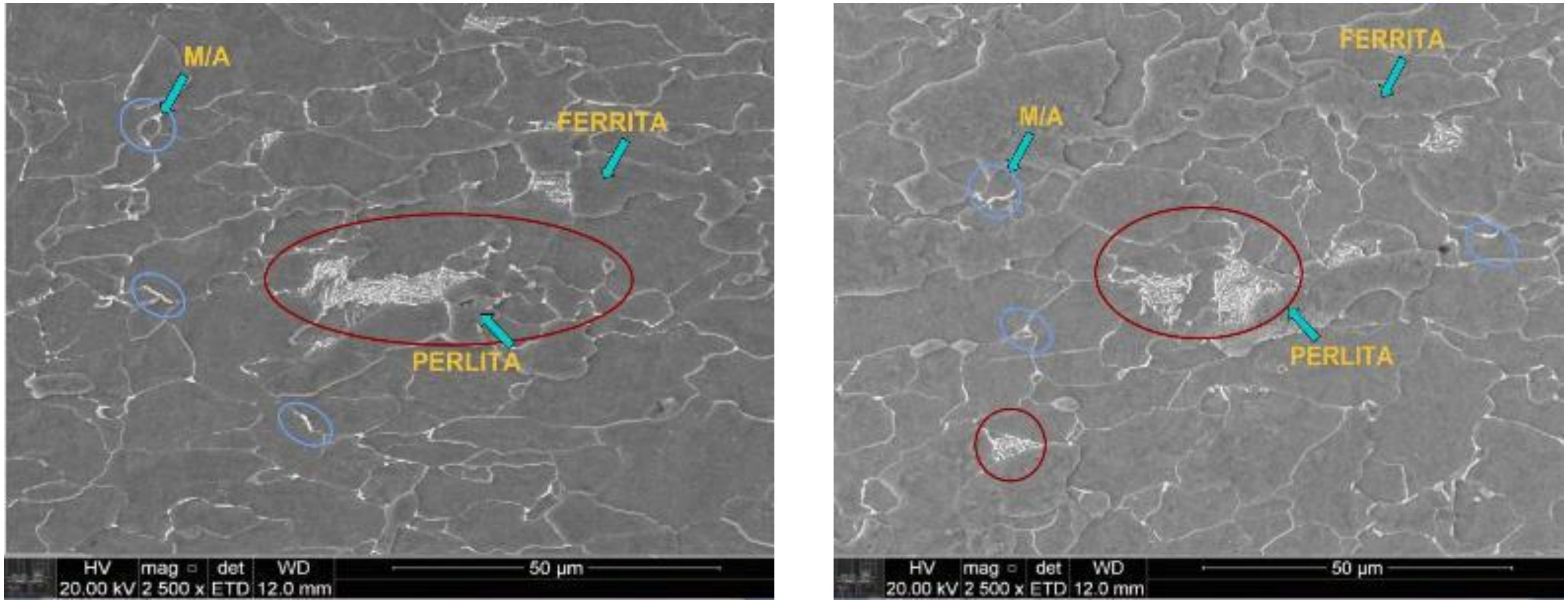


Figure - Optical microscopy of the central region of the steel cross section API 5L X65 LMn. Attack: Nital 2%.

Microestrutural characterization: API 5L X65LMn Steel



(a) (b)

Figure - Optical microscopy of the central region of the cross section (a) and longitudinal (b) of API 5L X65 LMn steel. Attack: Nital 2%. Grain size of medium 6.8 ± 0.3 µm, using circular intercepts method (ASTM INTERNATIONAL E112-13, 2014) with the help of the ImageJ program.

3. Microstructural characterization: A-36 Steel

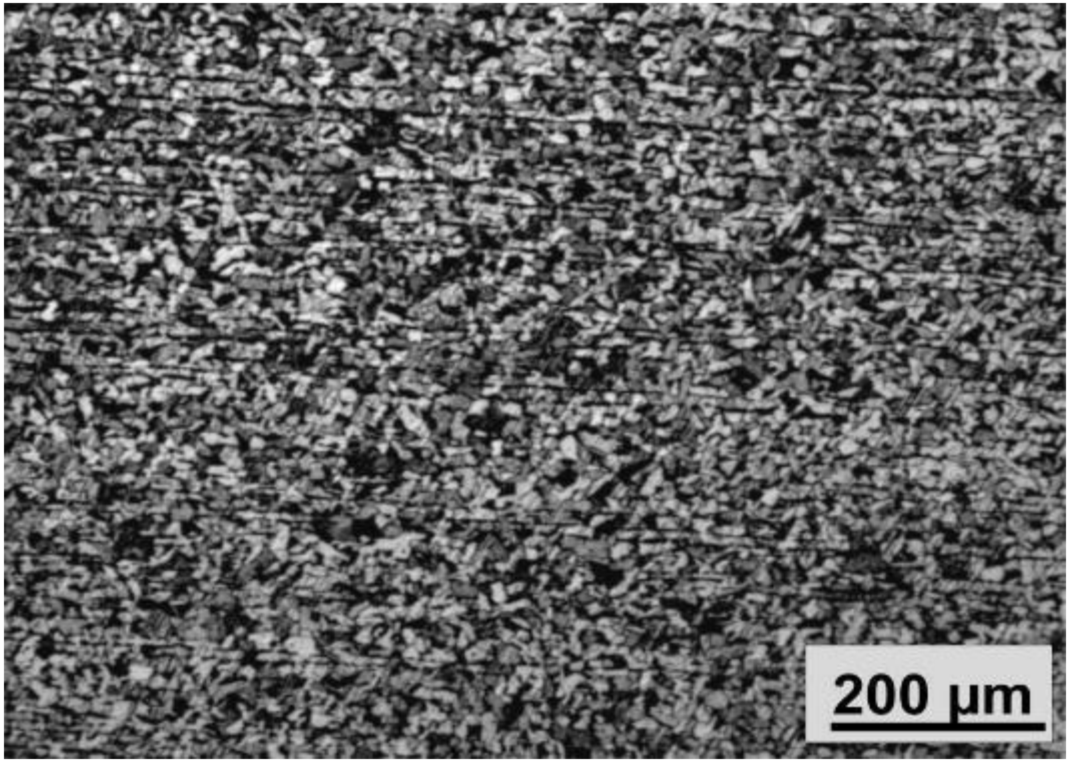
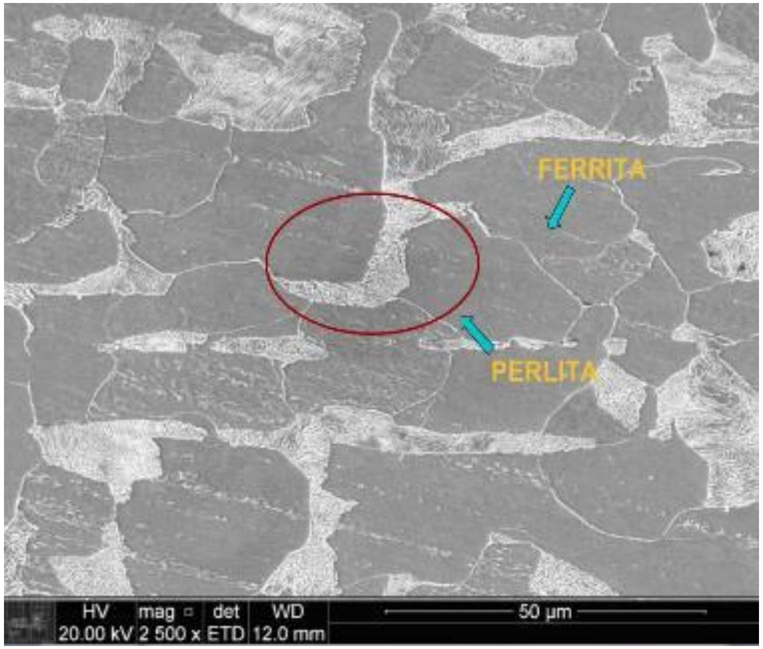
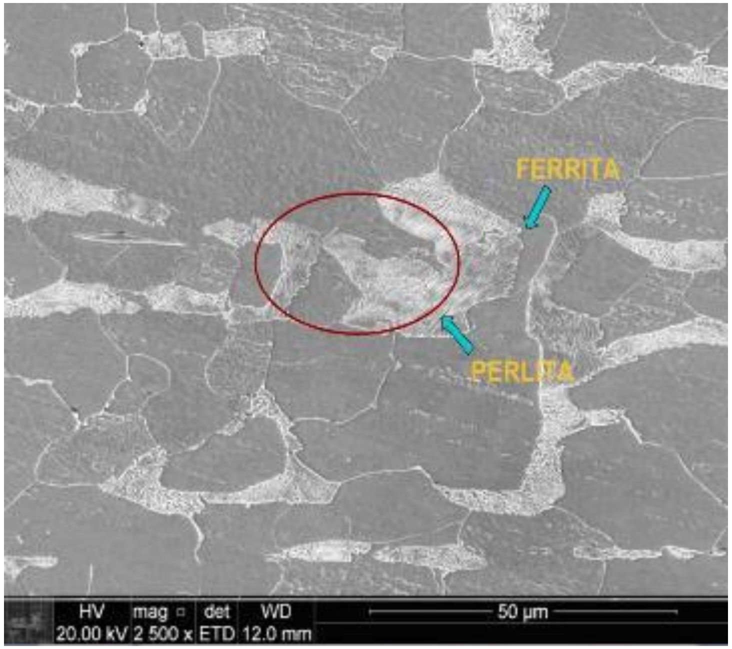


Figure - Optical microscopy of the central region of the steel cross section ASTM A-36. Attack: Nital 2%.

Microestrctural characterization: A-36 Steel



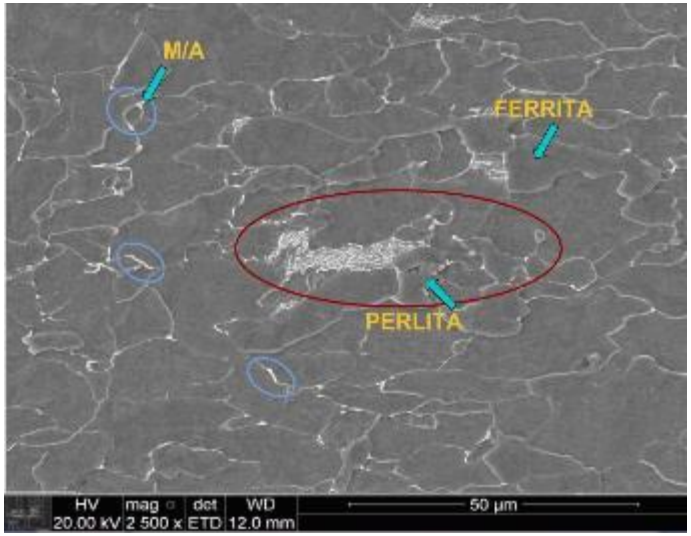


(a) (b)

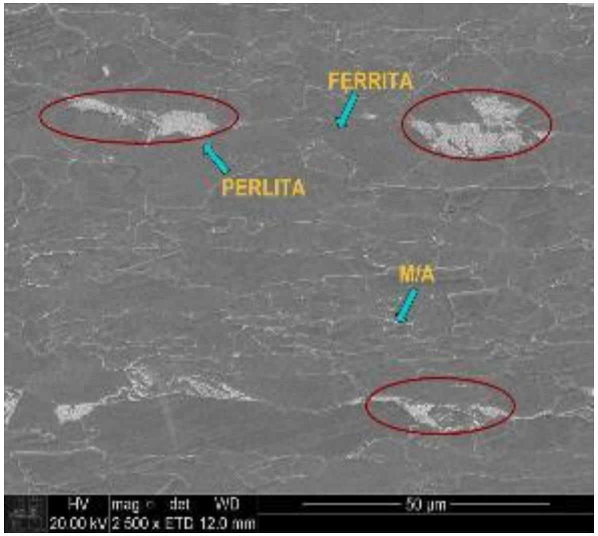
Figure - Optical microscopy of the central region of the cross-section (a) and longitudinal (b) of ASTM A-36 steel. Attack: Nital 2%. Grain size of medium 9.8 ± 0.3 µm, using circular intercepts method (ASTM INTERNATIONAL E112-13, 2014) with the help of the ImageJ program.

Steel microstructures

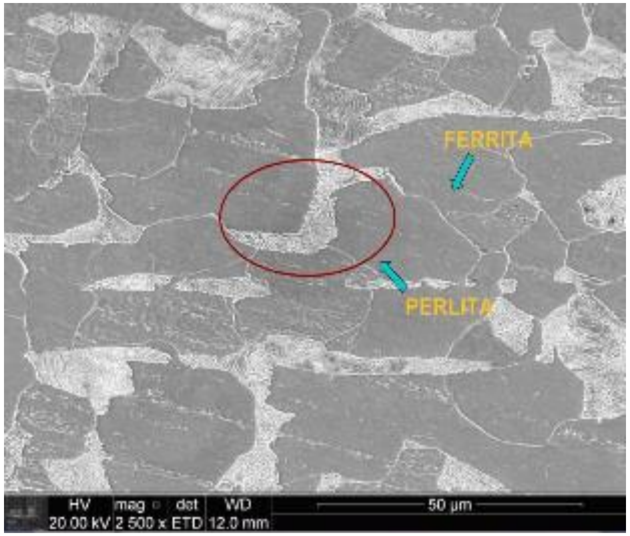
X6LMn



X65E



A36



III. Results of Electrochemical impedance spectroscopy (EIS)

1. Open circuit potential - OCP

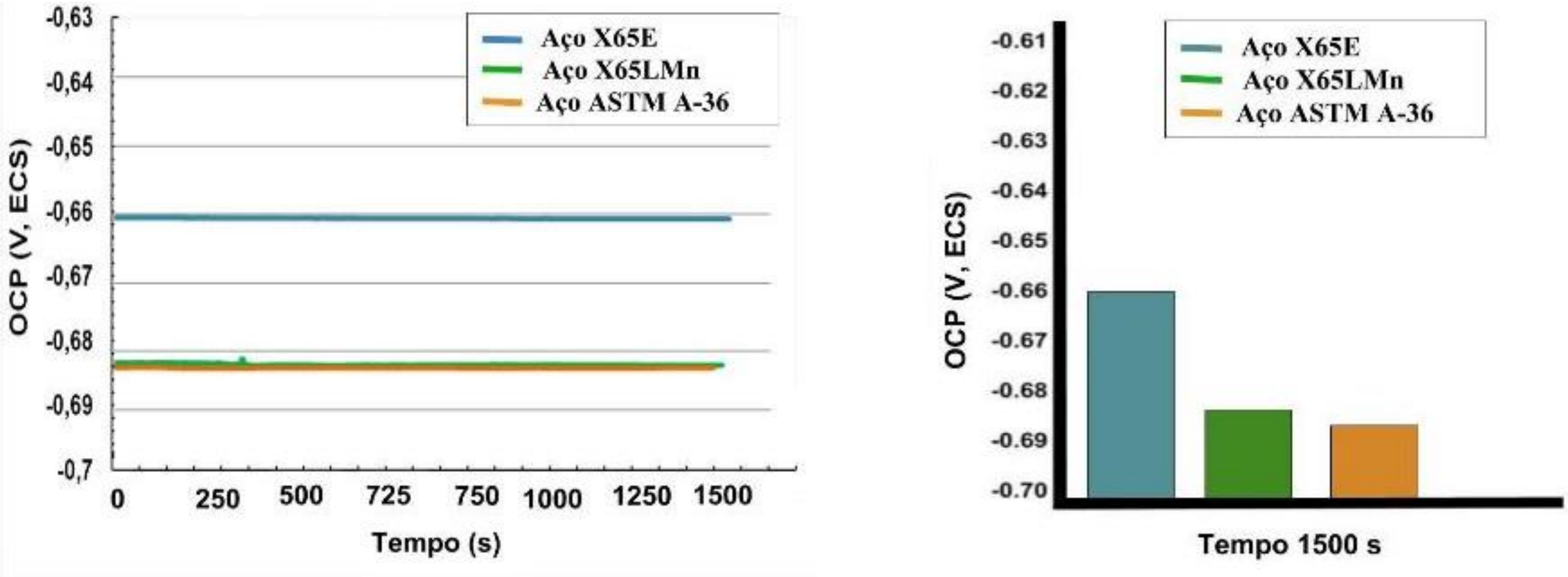


Figure - (a) Variation of the OCP as a function of the immersion time in a solution composed of deionized water, deaerated and CO2 saturation for the three steels. Total immersion time of 2h, (b) bar diagram variation of OCP.

1. Electrochemical impedance spectroscopy (EIS)

X 65E Steel

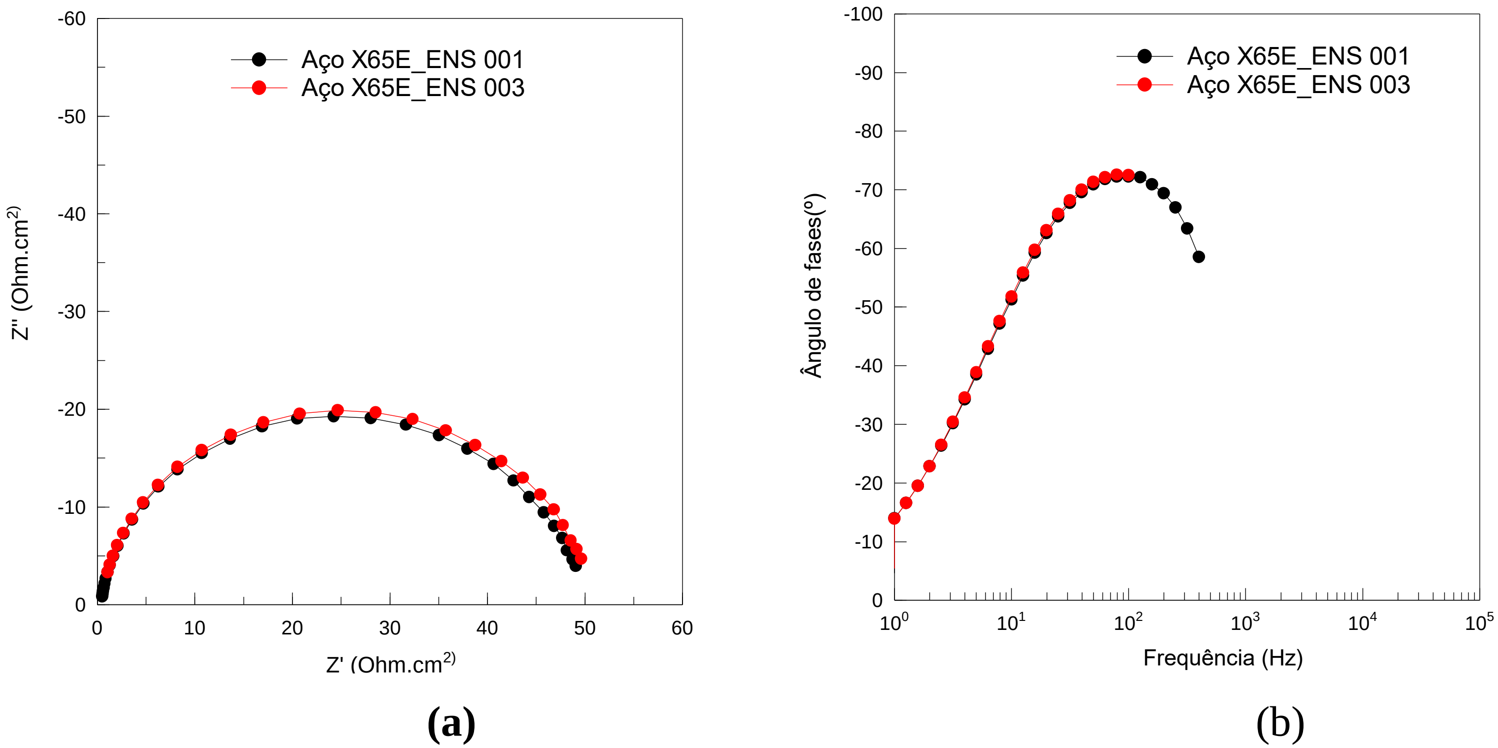
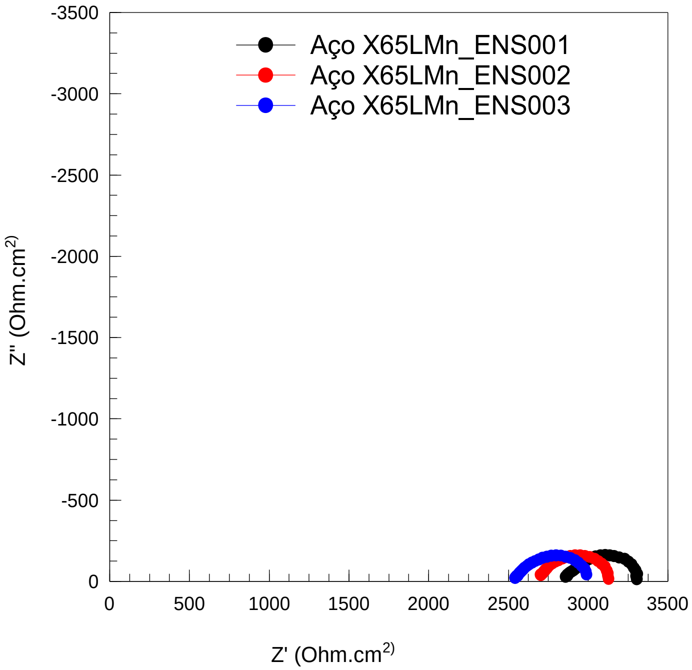
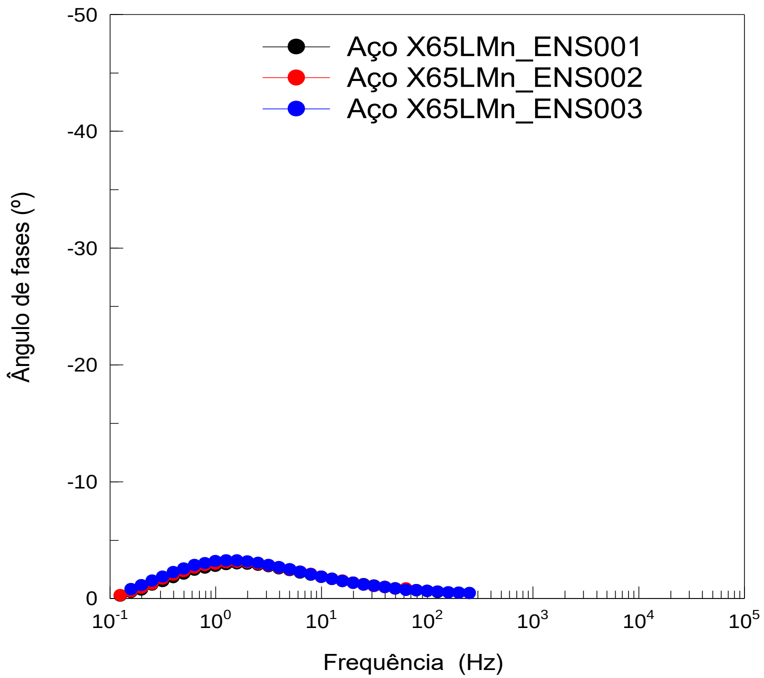


Figure - Nyquist (a) and phase angle (b) diagrams in solution composed of deionized water, deaerated and with CO_2 saturation for X65 E steel. Test 2

X 65LMn Steel



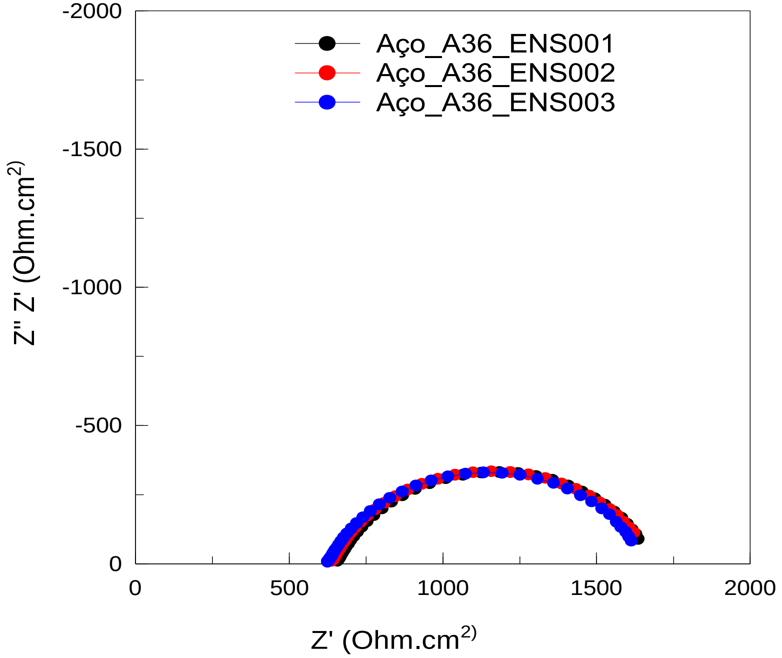
(a)



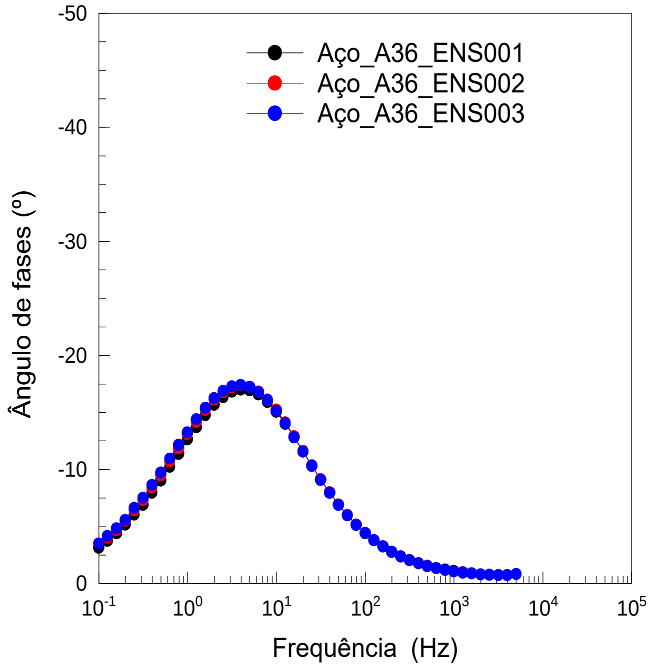
(b)

Figure – Nyquist plot (a) and phase angle (b) diagrams in solution composed of deionized water, deaerated and CO_2 saturated for X65 LMn steel.

A36 Steel



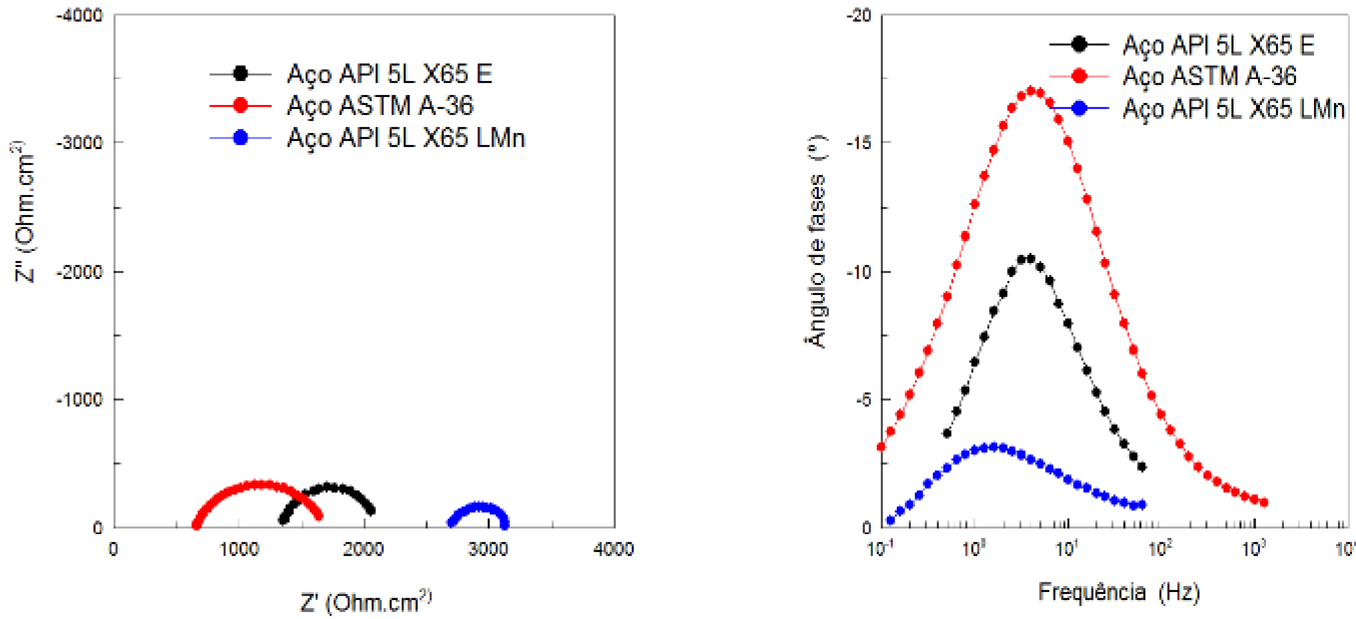
(a)



(b)

Figure - Nyquist (a) and phase angle (b) diagrams in solution composed of deionized water, deaerated and with CO2 saturation for the steel ASTM A-36

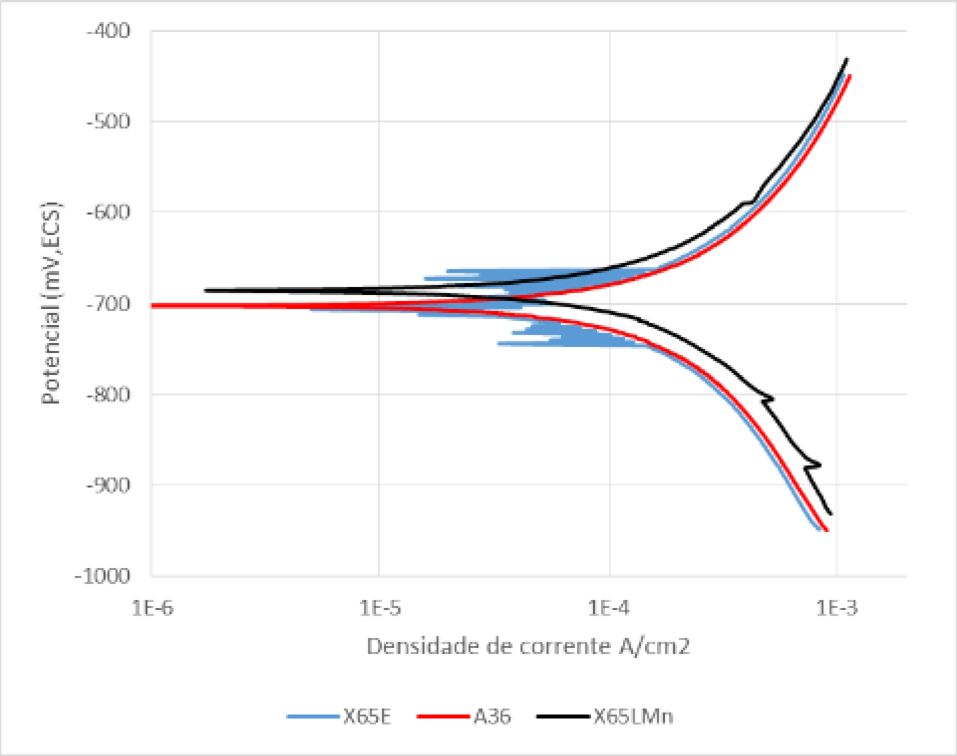
Comparing the steels



(a) (b)

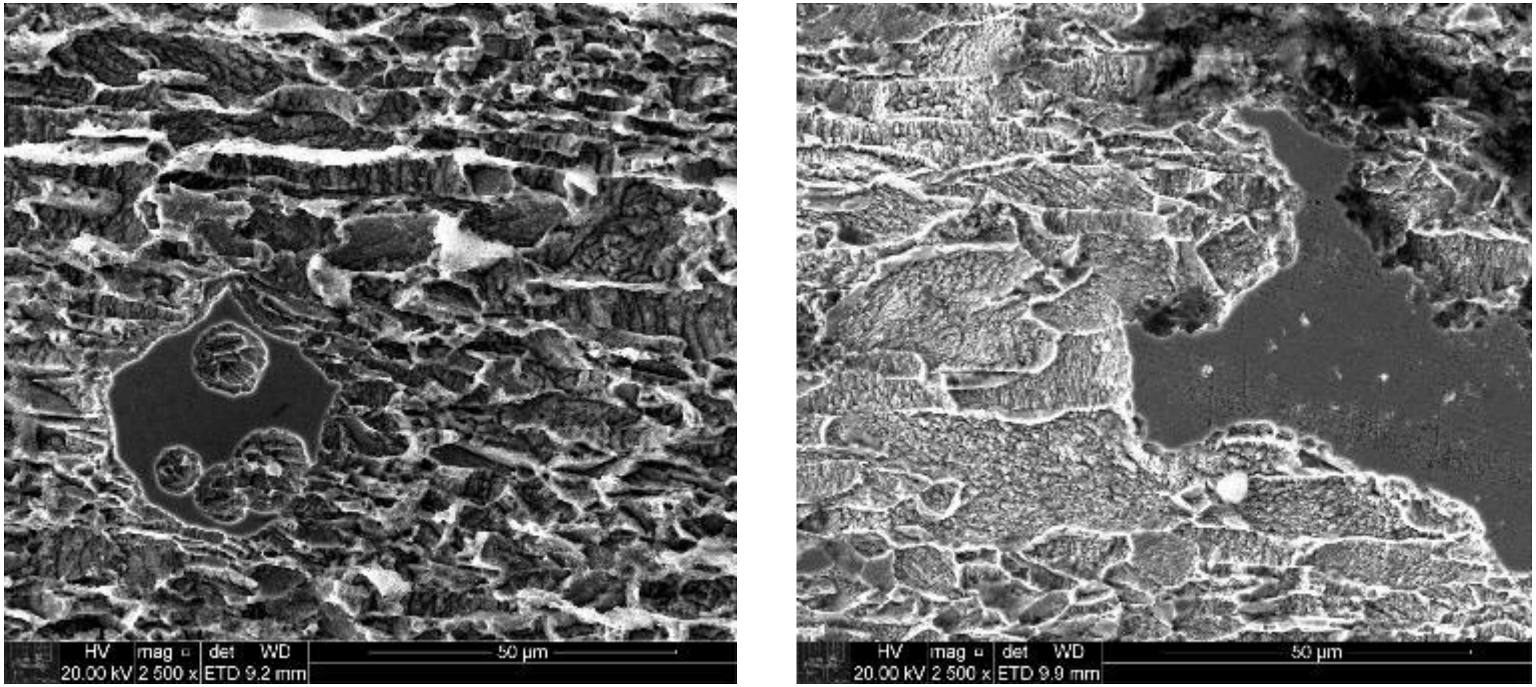
Figure - Nyquist (a) and phase angle (b) diagrams in solution composed of deionized water, deaerated and with saturation of CO_2 for the three steels

Potentiodynamic polarization results



IV. ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY RESULTS

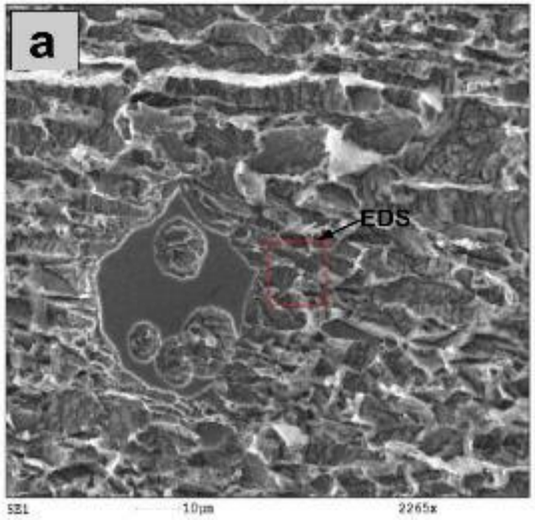
1. Corrosion morphology: X65E Steel



(a) (b)

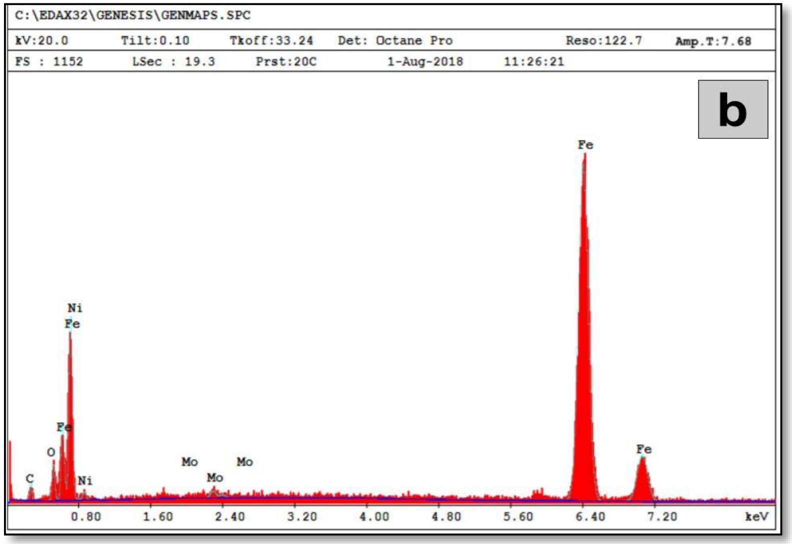
Figure - Image of corrosion morphology at 3 days (a) and 5 days (b) in solution composed of deionized water, deaerated and saturated with CO2.

3 days

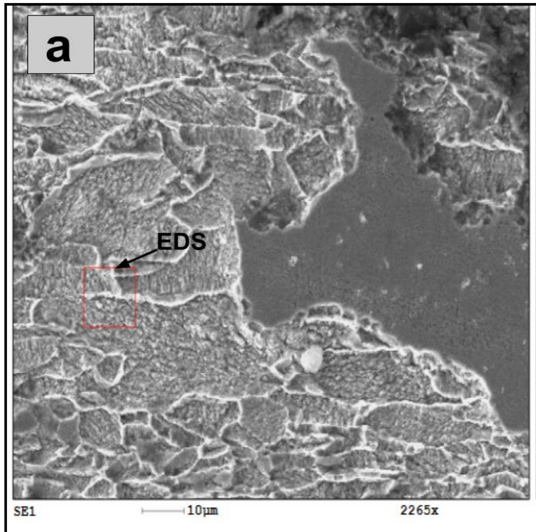


EDS Quantitative Results

Element	Wt%	At%
CK	3.73	14.40
OK	3.09	8.96
NiL	6.47	5.27
MoL	1.26	0.61
FeK	85.25	70.76

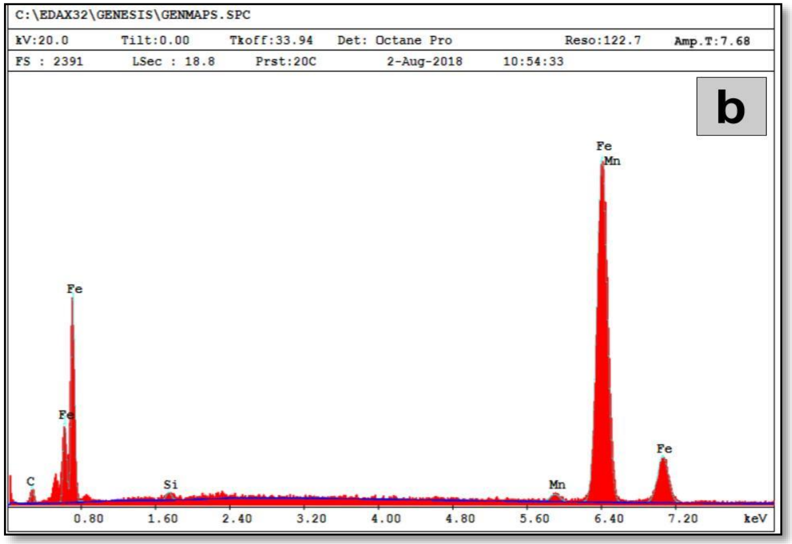


5 days

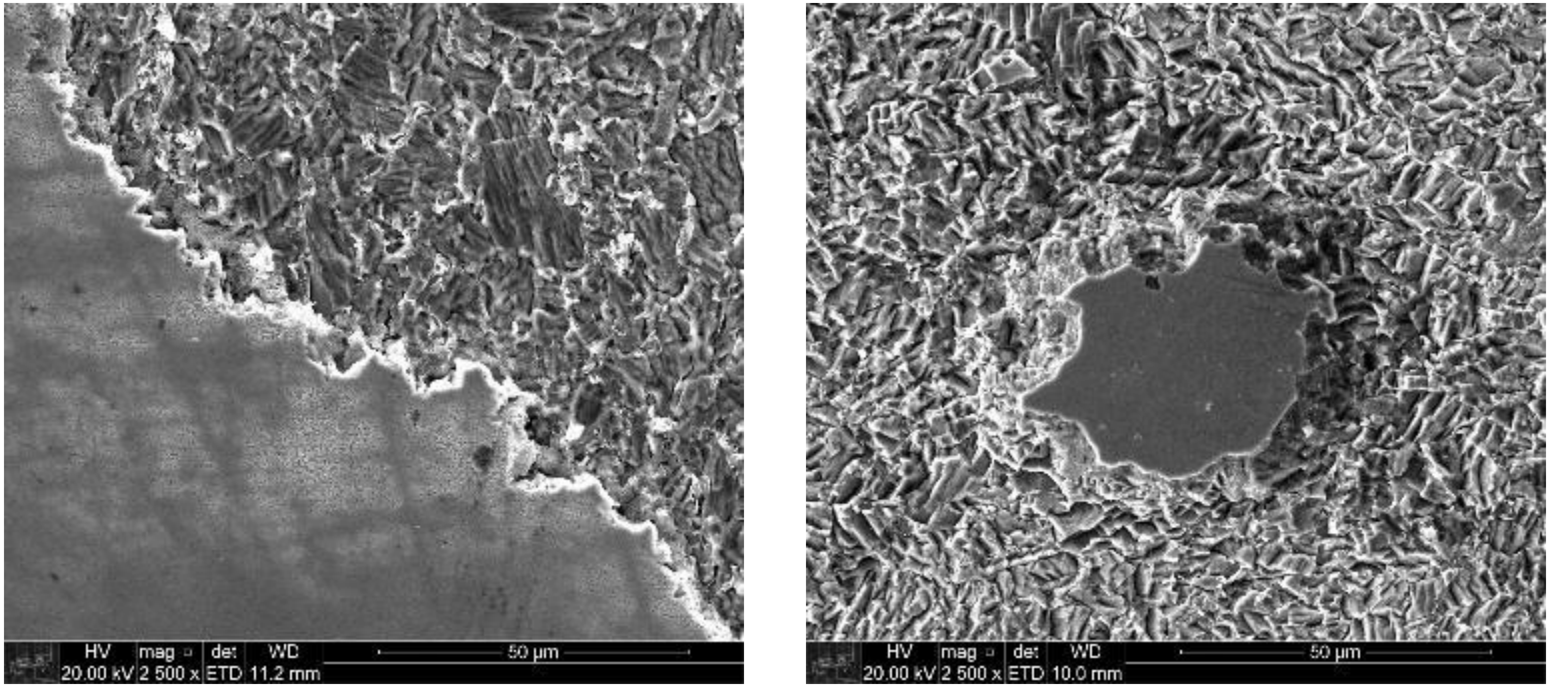


EDS Quantitative Results

Element	Wt%	At%
CK	4.17	16.73
SiK	0.67	1.15
MnK	1.71	1.50
FeK	93.46	80.63



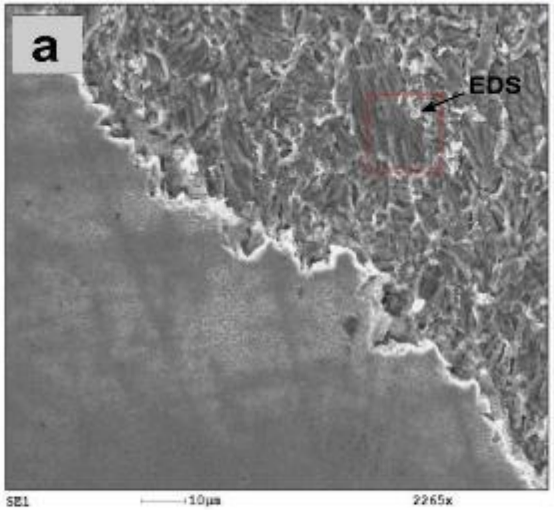
2. Corrosion morpholgy: X65 LMn Steel



(a) (b)

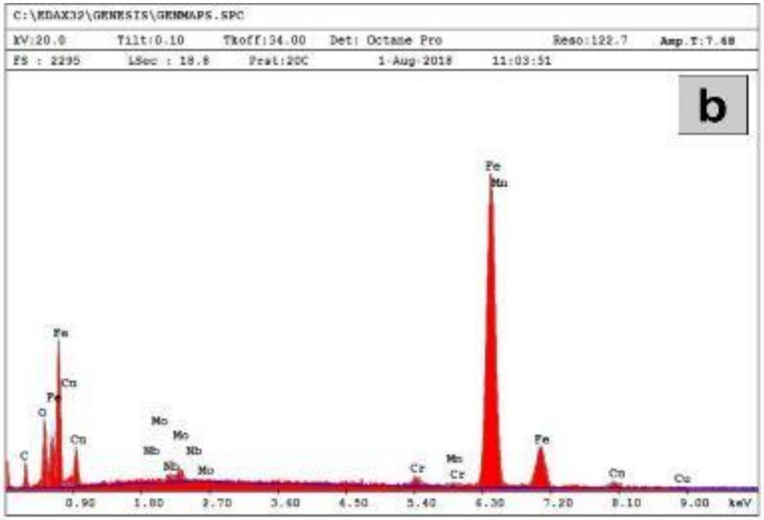
Figure - Image of corrosion morphology at 3 days (a) and 5 days (b) in solution composed of deionized water, deaerated and saturated with CO2.

3 days

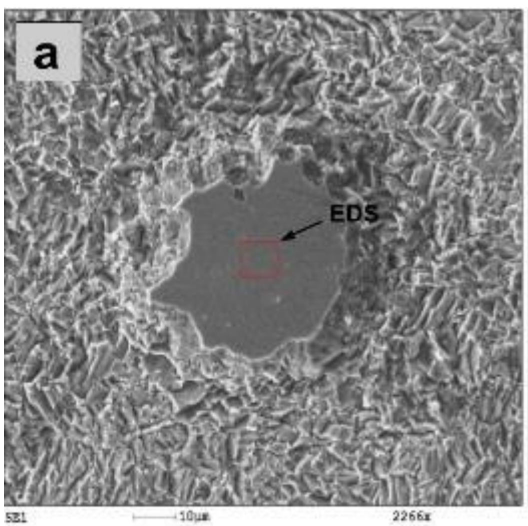


EDS Quantitative Results

Element	Wt%	At%
CK	5.81	19.94
OK	6.29	16.22
NbL	1.31	0.58
MoL	1.63	0.70
CrK	1.22	0.97
MnK	0.67	0.51
FeK	80.04	59.12
CuK	3.03	1.96

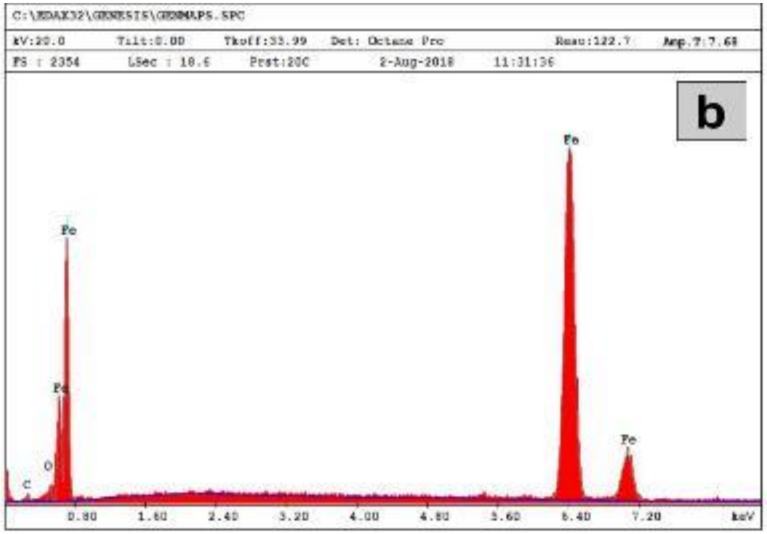


5 days

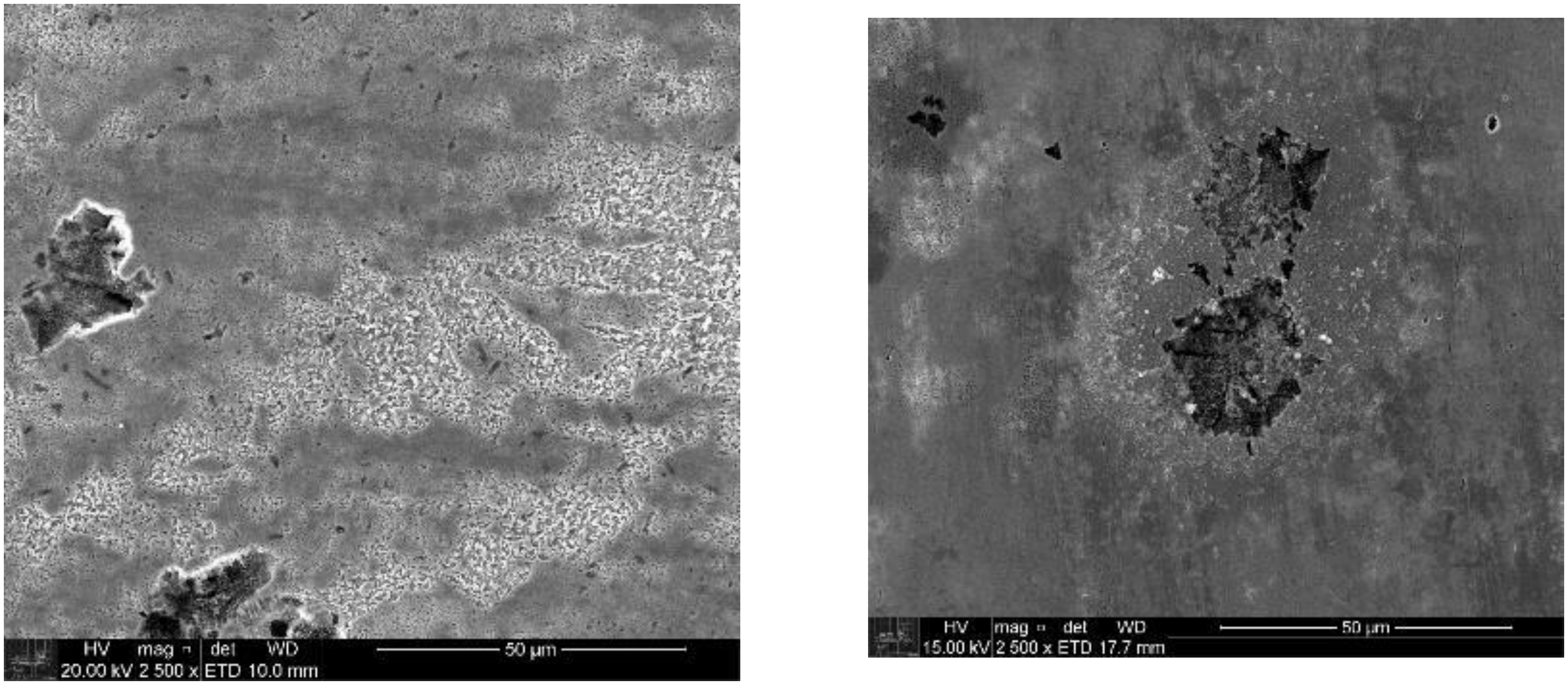


EDS Quantitative Results

Element	Wt%	At%
CK	1.76	7.43
OK	1.39	4.42
FeK	96.85	88.15



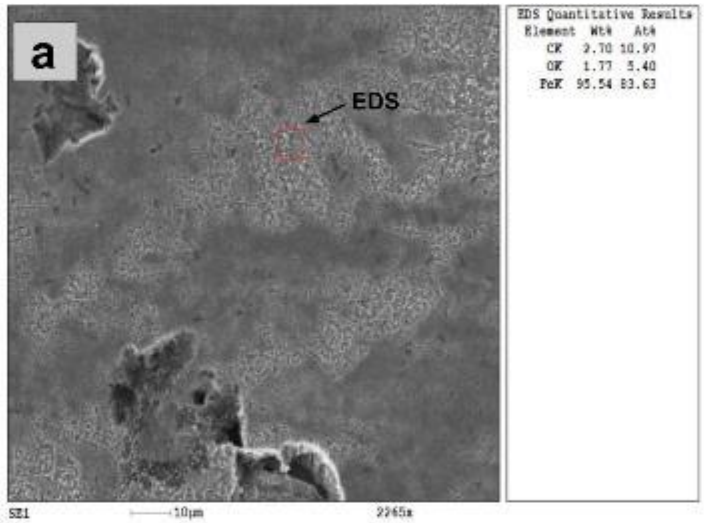
3. Corrosion morphology: A-36 Steel

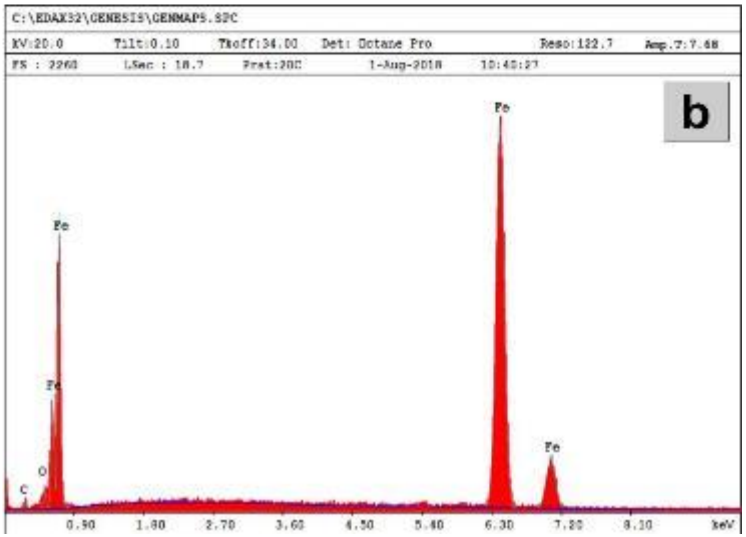


(a) 3 days (b) 5 days

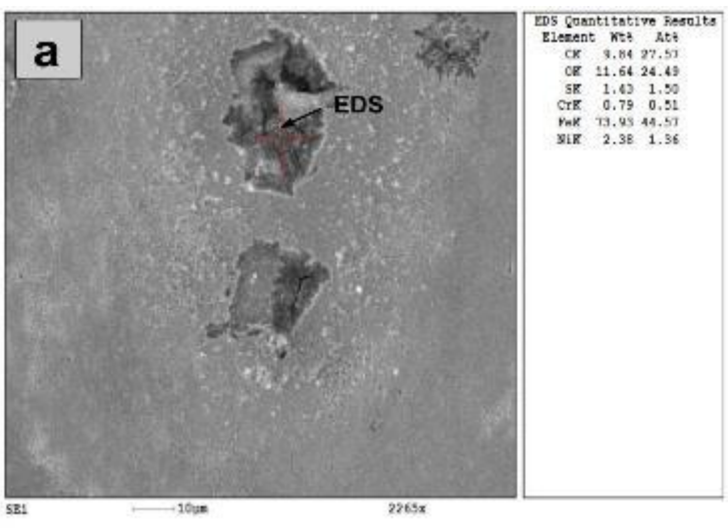
Figure - Corrosion morphology image at 3 days (a) and 5 days (b) in solution composed of deionized water, deaerated and saturated with CO2.

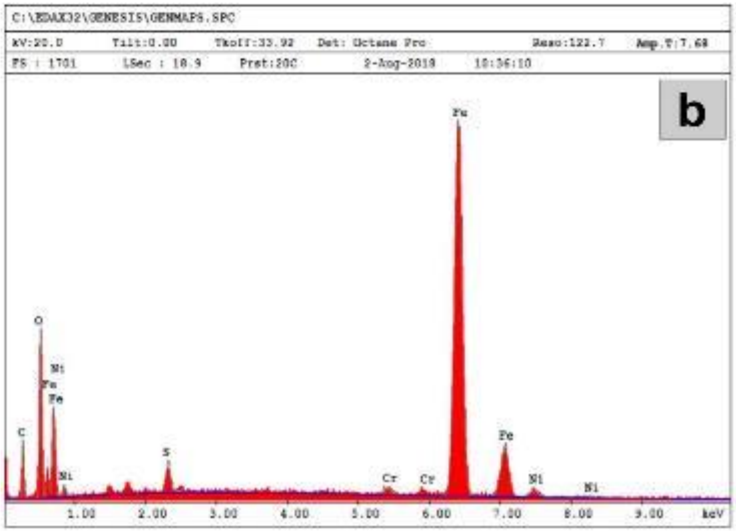
3 days


a
EDS
EDS Quantitative Results
Element Wt% At%
CK 2.70 10.97
OK 1.77 5.40
FeK 95.54 83.63
SE1
10µm
2265x

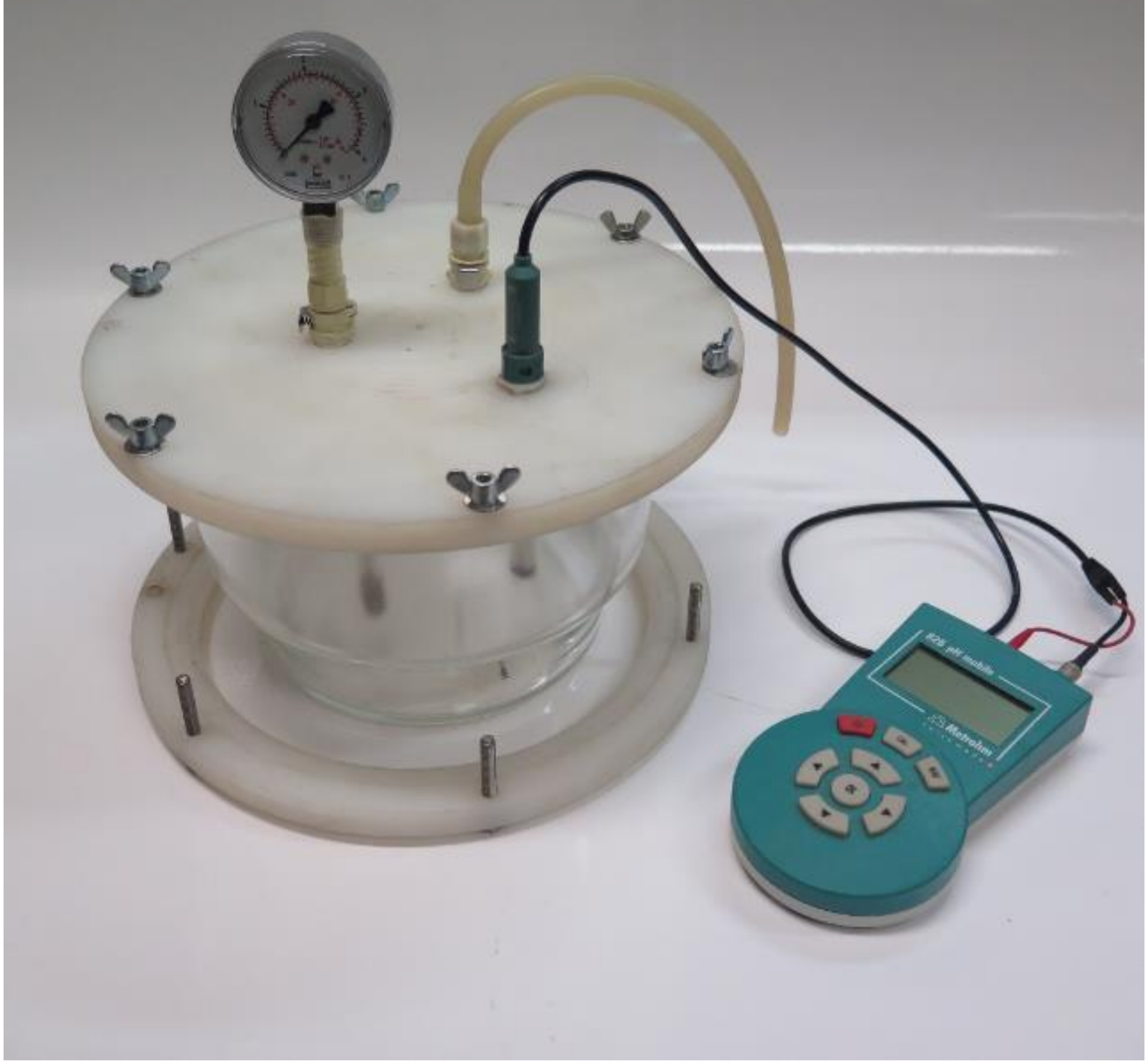

C:\EDAX32\GENESIS\GENMAPS.SPC
b
Fe
Fe
Fe
Fe
O
C
0.90 1.80 2.70 3.60 4.50 5.40 6.30 7.20 8.10 keV

5 days


a
EDS
EDS Quantitative Results
Element Wt% At%
CK 9.84 27.53
OK 11.64 24.49
SK 1.43 1.50
CrK 0.79 0.51
FeK 73.93 44.57
NiK 2.38 1.36
SE1
10µm
2265x


C:\EDAX32\GENESIS\GENMAPS.SPC
b
Fe
O
Ni
Fe
Fe
C
S
Cr
Cr
Fe
Ni
Ni
1.00 2.00 3.00 4.00 5.00 6.00 7.00 8.00 9.00 keV

Design of electrochemical corrosion cell



V. Weight loss tests

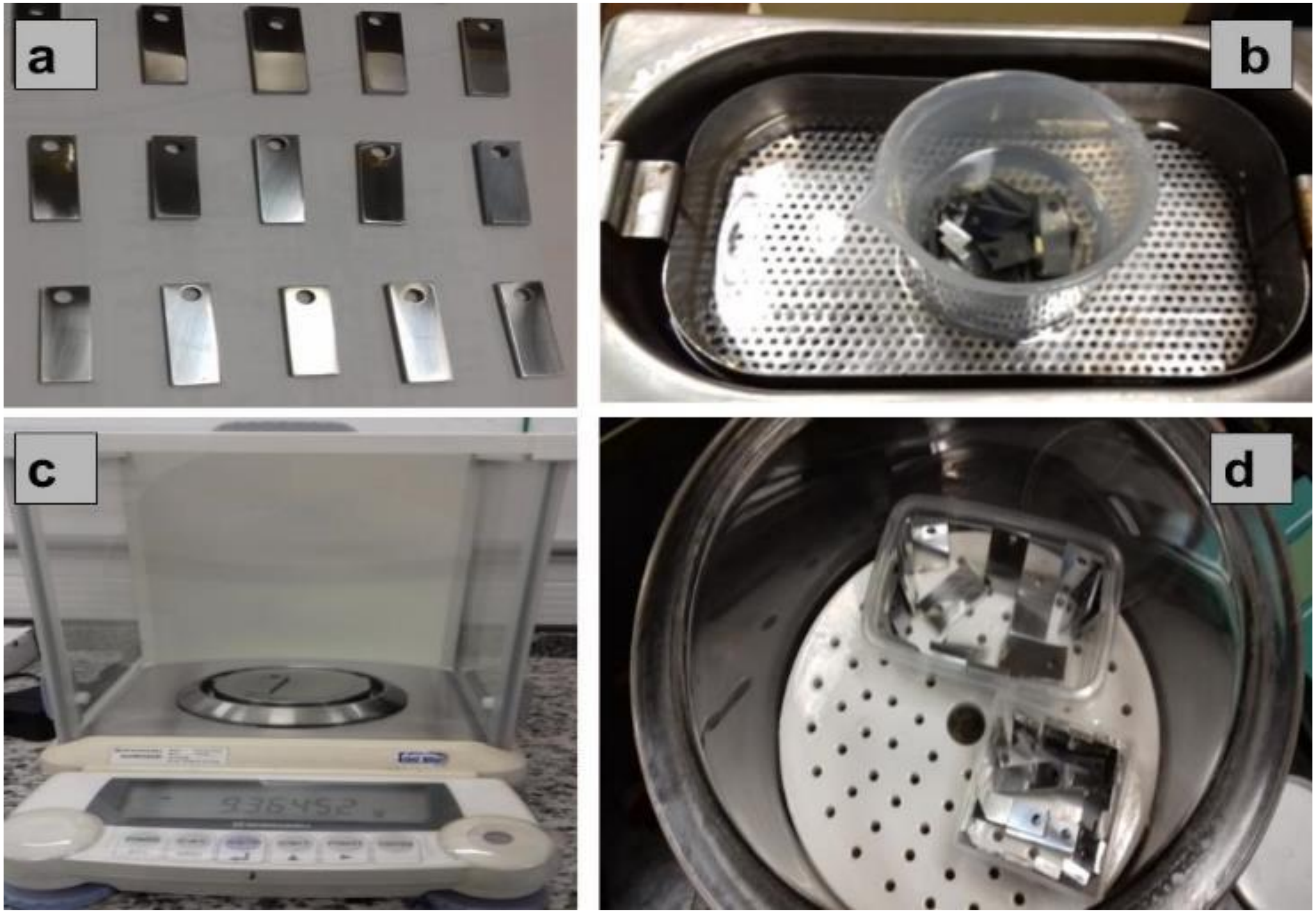


Figure. Experimental procedure for the weight loss tests

Aspect of the specimens after weight loss tests

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