

# Chemical, structural, mechanical and corrosion characterization of the extraction pipes of natural gas

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## Keywords

Microstructure, corrosion tests, mechanical tests

## 1. Introduction

Non-alloy steel pipe used in the extraction of natural gas components are part of C-Mn steels group [1] whose main chemical compositions and mechanical characteristics are presented in Table 1.

Table 1

Pipe no.	Steel mark	Chemical composition [%]			Mechanical characteristics		
		C	Mn	Si	R <sub>p0.2</sub> [N/mm <sup>2</sup> ]	R <sub>m</sub> [N/mm <sup>2</sup> ]	A <sub>5</sub> [%]
1	N80	0.33	1.65	0.33	664	836	19
2	J55	0.35	1.33	0.26	478	673	23
4	J55	0.28	1.34	0.21	456	658	24
5	N80	0.35	1.51	0.35	696	812	18
6	N80Q*	0.24	1.33	0.32	639	724	24
7	(Deleni)	Unknown					

\* hardness is max. 51HRC (~ 500 HV10)

The pipes dimensions for the expertise are:

- length: 1500 mm
- transversal section was:
  - Pipe 1 (N80) de: Ø 60.0 x 4.8 mm
  - Pipe 2 (J55) de: Ø 60 x 4.8 mm
  - Pipe 4 (J55) de: Ø 73 x 5.5 mm
  - Pipe 5 (N80) de: Ø 73 x 5.5 mm
  - Pipe 6 (N80Q) de: Ø 73 x 5.5 mm
  - Pipe 7 (Deleni) de: Ø 73 x 5.5 mm

## 2. Experimental program

The experimental program included tests such as examinations and mechanical tests, the values obtained are compared with the chemical characteristics, structural and mechanical values namely:

- chemical composition,
- metallographic structures,
- mechanical characteristics of strength and deformability,
- mechanical characteristics of tenacity,
- evaluation of tension corrosion in saline environment,
- evaluation of local aging phenomena,
- evaluation of plastic deformation phenomena to compression by flattening.

## 3. Chemical characterization

Chemical compositions of the pipes subject to expertise program were determined with Innovix - Systems equipment from ISIM Timișoara, on the exterior surfaces and the experimental results obtained are inserted in Table 2.

Chemical variations on the six pipes are presented in Figure 1.

Table 2

Pipe no.	Chemical compositions [%]					
	C <sub>m</sub> *	Mn	Si	Cr	Ni	V
1	0.32	1.49	0.48	0.05	---	---
2	0.36	1.22	0.48	0.08	---	---
4	0.28	1.15	0.48	0.07	0.10	---
5	0.32	1.10	0.52	0.04	---	0.17
6	0.25	1.33	0.47	0.05	---	0.16
7	0.32	1.24	0.46	0.05	0.13	---

\*carbon determined with the phases and constituents relation (informative values) [2]

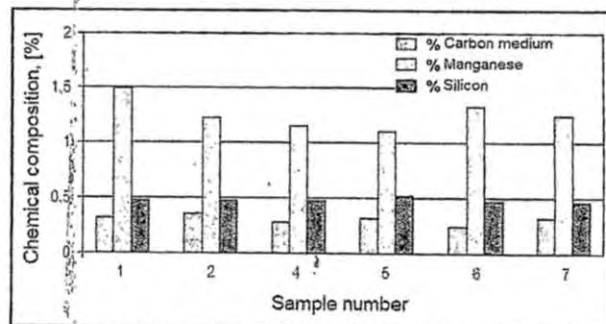


Figure 1. Elements variation on the analyzed pipes

Analyzing the variations of the main chemical elements it is observing:

- manganese percentages are below those indicated in the quality certificates
- silicon percentages values are higher those of the quality certificates
- average percentage of carbon determined by phases and constituents relation is similar to those of the pipe from the quality certificates.

## 4. Structural characterization

Pipe samples M1, M2, M4, M5, M6 and M7 with length of 50 mm were taken from the analyzed pipes for the

microstructural evaluation on the pipe thickness. The microstructures determined were:

- At sample M1, pearlite-ferritic microstructure with the grain score of 7-8 according to ISO 643: 2003, the pearlite percent does not exceed 42% (Figure 2);



Figure 2. Sample M1, BM, [Etched Nital 2%, 100x]

- At sample M2, microstructures consisting of pearlite and ferrite, the grain score is between 6-8, according ISO 643: 2003, the pearlite percent is max. 45% (Figure 3).

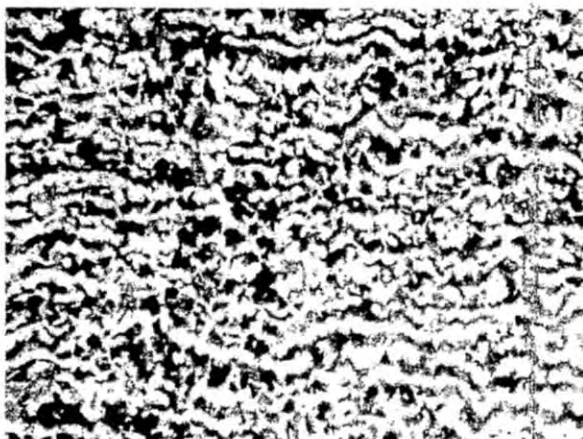


Figure 3. Sample M3, BM [Etched Nital 2%, 100x]



Figure 4. Sample M4, BM [Etched Nital 2%, 100x]

- The sample M4 presents pearlite-ferritic microstructures with the grain scores between 6 and 8 according to ISO 643: 2003 the pearlite percent is 35% (Figure 4).

- At sample M5, microstructures consisting of pearlite and ferrite the pearlite grain score is between 3 and 7 according to ISO 643: 2003 the pearlite percentage is 40% (Figure 5).

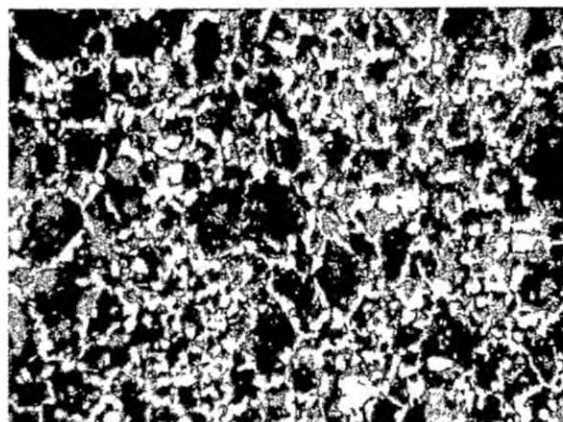


Figure 5. Sample M5, BM [Etched Nital 2%, 100x]

- At sample M6, pearlite-ferrite microstructures with the pearlite grain scores between 3 and 7 according to ISO 643: 2003, the pearlite percentage does not exceed 35% (Figure 6).

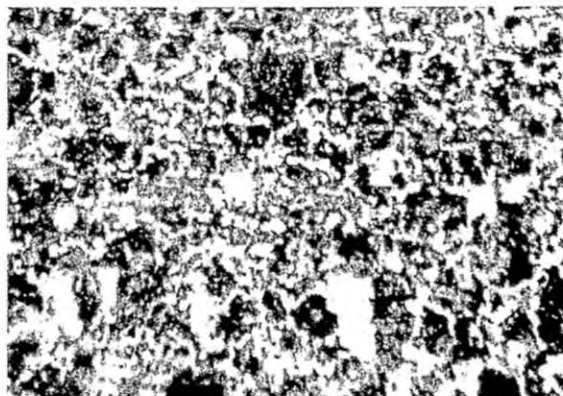


Figure 6. Sample M6, BM [Etched Nital 2%, 100x]

- At sample M7 pearlite-ferrite microstructure, the pearlite grain scores ranging between 3 and 7 according to ISO 643: 2003, and the pearlite grains does not exceed 40% (Figure 7).

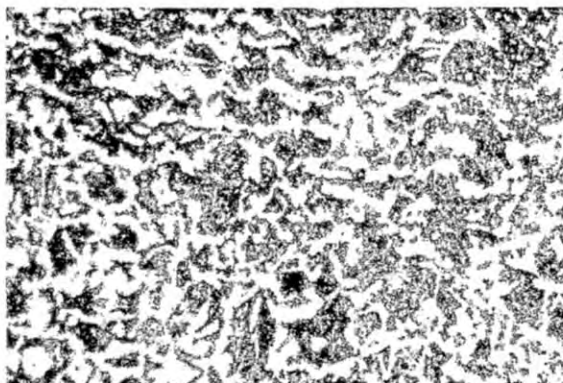


Figure 7. Sample M7, BM [Etched Nital 2%, 100x]

In the examined surfaces of the pipes were not detected defects such as microcracks.

## 5. Mechanical characterization

### 5.1. HV10 Hardness test

HV10 hardness test was performed on the pipes thickness according to EN ISO 6507-4: 2005. In table 3 are presented the maximum and minimum hardness values and the values of  $\Delta HV10$  structural hardening estimator, calculated with the relation (1).

$$\Delta HV10 = \frac{HV10_{max} - HV10_{min}}{HV10_{max}} \cdot 100 \quad [\%] \quad (1)$$

where:

- $HV10_{max}$  is the maximum hardness HV10 in one zone of the transversal section,
- $HV10_{min}$  is the minimum hardness HV10 in another zone of the transversal section.

It is considered that, if  $\Delta HV10 \geq 50\%$ , in the analyzed zones there were developed embrittlement phenomena and there is a high risk of brittle fracture apparition.

Table 3

Pipe no.	HV10 hardness		$\Delta HV10$ Estimator [%]
	Minimum value	Maximum value	
M1	276	283	2.41
M2	207	209	4.30
M4	196	207	5.71
M5	285	297	4.04
M6	283	294	3.74
M7	228	264	13.63

Analyzing  $\Delta HV10$  estimator values of the six pipes analyzed, it is observing that the maximum value is 13.63%, well below the critical value of 50%, attesting that have not developed accentuated local hardening phenomena on the pipes thickness made of C-Mn steel alloy.

### 5.2. Tensile tests

Tensile tests performed on two specimens taken from pipe 7 respecting the conditions of EN 895: 1997 highlighted the mechanical characteristics:

- yield strength between 388 and 396 N/mm<sup>2</sup>,
- tensile strength between 620 and 612 N/mm<sup>2</sup>,
- elongation of max. 26%,
- reduction area of max. 63%.

These mechanical characteristics are specific to unalloyed C-Mn steel and the mechanical characteristics ( $R_{p0.2}$ ,  $R_m$ ) are lower than other steels examined.

### 5.3. Bending tests

Bending test performed according to EN 5173: 2010, on flat specimens using a 80 mm diameter tool revealed no cracks on bent surfaces to 180°, evidencing a high capacity of plastic deformation of the analyzed materials.

### 5.4. Impact bending test

Impact bending test according to EN 875: 1997 KV was performed on flat specimens at room temperature (23°C) and

the fracture energy values  $KV_0$  are presented in the graph from Figure 8.

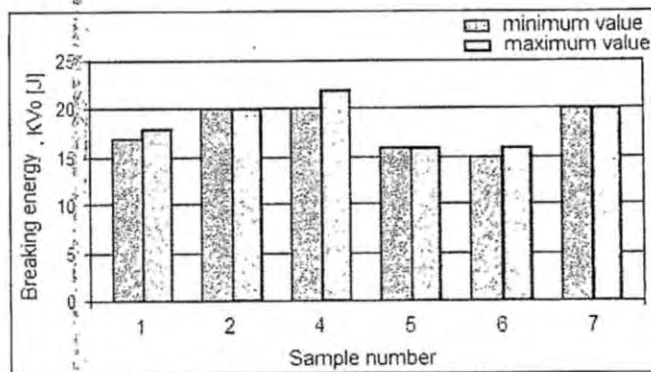


Figure 8. Variation  $KV_0 = f(\text{pipe})$

Analyzing the fracture energy values is observed that no value reaches the min. 27 J at 20°C accepted for alloyed and non-alloyed pipes used in gas extraction industry and petroleum products according to EN 10208-2:1999 and EN 10216-2: 2003.

### 5.5. Tensothermal embrittlement test

The tensothermal embrittlement trend [3] of non-alloy steel of construction pipes used for the extraction of natural gas by determining the rupture energy  $K_I$ , was evaluated on resilience specimens plastic deformed with 7% and subjected to further heating for one hour at 250°C, according to STAS 56 774: 79. The summarized experimental results are listed in Table 4, where also were inserted and the aging degree values,  $G_I$ .

$$G_I = \frac{KV_0 - KV_I}{KV_0} \cdot 100 \quad [\%] \quad (2)$$

where:

- $KV_0$  is the maximum breaking energy determined in the actual state (0)
- $KV_I$  is the minimum breaking energy value determined in the tensothermal embrittlement state (I)

It is considered that the analyzed materials do not present

Table 4

Pipe no.	Breaking energy, $KV_I$ [J]		Embrittlement degree, $G_I$ [%]
	Minimum value	Maximum value	
1	14	15	22.2
2	15	16	25.0
4	15	17	31.8
5	14	15	12.5
6	13	14	18.75
7	15	16	25.0

tensothermal embrittlement sensitivity if  $G_I \geq 50\%$ . Variation of the embrittlement degree  $G_I$  of analyzed pipes 1, 2, 4, 5, 6, 7 is shown in Figure 9.

Analyzing  $G_I = f(\text{pipe})$  variation it is observed that the maximum embrittlement degree  $G_I$  is 31.8% at pipe 4 and the minimum value is 12.5% at pipe 5, thus there is a group "A" of embrittlement increasing from the pipe 1 to pipe 4 and a



group "B" of embrittlement increasing from pipe 5 to pipe 7. Group A contain two pipes with the outside diameters of

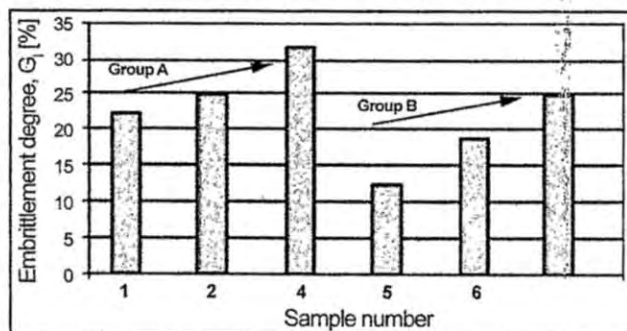


Figure 9. Variation  $G_1 = f(\text{pipe})$

$\varnothing 60$  and  $\varnothing 73$  mm and group B only pipes with the outside diameter of  $\varnothing 73$  mm.

### 5.6. Stress-corrosion testing

Stress-corrosion testing were made on two bending samples which were bent to  $180^\circ\text{C}$  followed by a immersion in saline medium (150 g NaCl la 1000 ml water) for 310 hours [4]

The tests were made in a plastic container that was continuously stirred so that the test specimens are covered with saline solution.

Before and after testing, all specimens were weighed for the assessment of mass loss ( $\Delta M$ ) and to determine the corrosion rate ( $C_R$ ) using the relationship:

$$C_R = \frac{\Delta M}{t} \text{ [g/h]} \quad (3)$$

where:

- $\Delta M$  is the mass loss ( $M_0 - M_f$ ),
- $t$  is the effective time for stress-corrosion testing.

Table 5.

Sample mark	Bending force [N]	Mass [g]		Mass loss, $\Delta M$ [g]	Corrosion rate, $C_R$ [g/hour]
		$M_0$ (initial)	$M_f$ (final)		
1.1	8,950	155	152	3	0.0096
1.2	12,680	155	151	4	0.012
2.1	6,700	150	147	3	0.0096
2.2	8,220	155	150	5	0.016
4.1	8,880	160	155	5	0.016
4.2	10,400	165	158	7	0.022
5.1	10,000	150	147	3	0.0096
5.2	11,500	150	146	4	0.012
6.1	10,550	155	150	5	0.016
6.2	11,100	155	151	4	0.012
7.1	8,090	160	155	5	0.016
7.2	8,450	155	152	3	0.0096

The experimental results obtained are presented in Table 5. The aspect of tested specimens in saline environment is shown in Figures 10÷12.

Corrosion rates (CR) of the tested samples have value between 0.0096 and 0.022 g/h while the mass loss during the corrosion test does not exceed 7g.

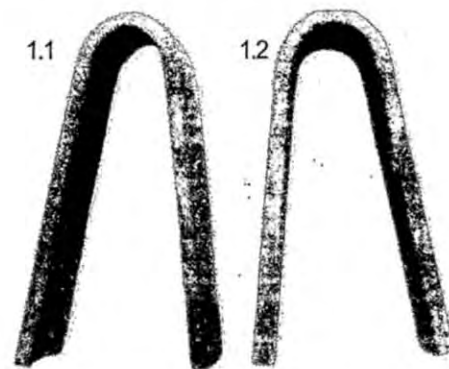


Figure 10. Tested samples 1.1 and 1.2

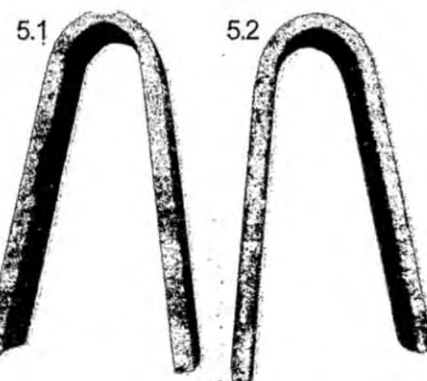


Figure 11. Tested samples 5.1 and 5.2

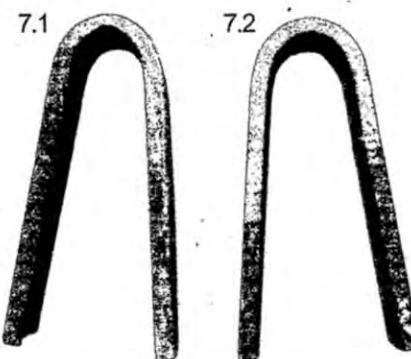


Figure 12. Tested samples 7.1 and 7.2

On the bent surfaces of the specimens tested for corrosion have not appeared cracks.

### 5.7. Flattening tests

Flattening tests according to SR EN ISO 8492: 2005, were made on samples taken from the pipe of 100 mm. All the tested samples showed lateral cracks at plastic in compression test machine (Figure 13÷16).

The presence of cracks on the side surfaces of the flattened pipes show a low capacity of the non-alloy steel to compression deformation.

## 6. Conclusions

6.1. Analyzed steel pipes are part of unalloyed steels C-Mn which carbon percentage does not exceed 0.35 and manganese percentage does not exceed 1.65.



Figure 13. Flattening sample 1



Figure 14. Flattening sample 2



Figure 15. Flattening sample 6

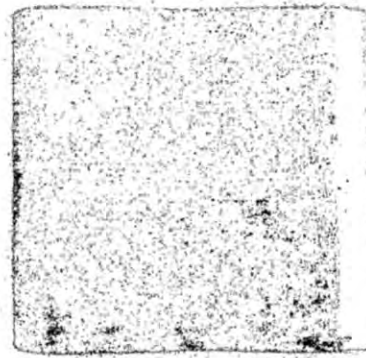


Figure 16. Flattening sample 7

6.2. The steel microstructures are pearlite-ferrite with the hardness between 200 and 297 HV10.

6.3. Mechanical characterization of analyzed pipes was based on the application of mechanical tests (tensile, bending shock compression flattening) that allowed the evaluation of mechanical characteristics of alloyed C-Mn steel on an experimental program agreed with the customer.

6.4. Characterization of stress-corrosion test in saline environment showed corrosion rates ranging between 0.0096 g/h and 0.022 g/hour, acceptable values for an accelerated corrosion test. Bent samples have cracked during the test.

6.5. Resistance characteristics of non-alloy steel pipe 7 are lower than the category steel used to manufacture the others pipes

6.6. Pipe tenacity is considered low, below 27 J at 20°C required by the standard SR EN 10208 - 2: 1999.

6.7. Characterization of tensothermal embrittlement shows that materials do not presents a pronounced sensitivity to aging; the degree of aging is well below 50%.

6.8. At all C-Mn steels appear aging trends occurring both on A direction at specimens 1, 2, 4 with different diameters ( $\varnothing 60$  mm and  $\varnothing 73$  mm) and on the direction B at specimens 5, 6, 7 which have the same diameter ( $\varnothing 73$  mm).

## References

- [1] \* \* \*: Certificate de calitate pentru oțeluri nealiate de tip C-Mn, Anexa 1, contract C 57/1, ISIM, 2011, Timișoara, România
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