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MICROSTRUCTURE EXAMINATIONS IN CORNERS OF THE LOW-CARBON STEEL SLABS FROM CONTINUOS CASTER MACHINE

The exposed selvedge layers in slabs cast by the continuous process should be free from surface defects, which in most cases appear in the form of cracks on the casting surface and run to its interior. In addition to the parameters of the casting process, the occurrence of such defects depends on the chemical composition of cast steel, on the segregation of surface active elements and formation of the precipitates of carbides, nitrides and other phases. Due to the frequent occurrence of defects in corners of the slabs, non-destructive testing was performed on the mechanically cleaned surfaces of slabs. The test material was low-carbon API(American Petroleum Institute API 5L standard) steel micro alloyed with Nb and Ti designed for the production of pipes to handle gas, oil and other liquid and gaseous fuels. Despite the use of different methods of inspection, i.e. ultrasonic, magnetic particle and penetrant, cracks were not traced in the examined material. Then, from the corners of the examined slabs, specimens were cut out for metallographic examinations. The main purpose of these examinations was to disclose the presence of possible cracks and micro cracks on the surfaces transversal and longitudinal to the direction of casting. At the same time, studies were conducted to establish the number and morphology of non-metallic inclusions in selvedge layers of the slab corners and axis. Additionally, hardness of the slabs was measured. The conducted studies revealed only some minor differences in the slab hardness along its axis ($130 \div 135$ HB) and in selvedge layers ($120 \div 123$ HB) . *Keywords*: API 5L line pipe steels, Low-carbon micro alloyed steel, Microstructure, Non-metallic inclusion

1. Introduction

Low-carbon micro alloyed steels in grades L360MB and L360MBS belong to the group of API steels designed for pipes handling liquid fuels and gas in the mining, chemical and petrochemical industries $[1\div 4]$. The use in these steels of micro alloying elements like Nb, V and Ti leads to grain refinement and microstructure strengthening during controlled rolling with the resulting precipitation of fine-dispersed carbides and carbonitrides [5,6]. The result is a favourable combination of mechanical and plastic properties offered by these steels [4,5,7,8]. Low content of gas and harmful impurities (P and S) allows maintaining a low level of the non-metallic inclusions, whose amount, type and morphology are of paramount importance for the steel cleanliness [9]. Non-metallic inclusions are formed in various stages of the steel preparation for continuous casting. Most frequently, in steels deoxidized with aluminium, oxide inclusions (Al₂O₃) are observed to occur as well as sulphides (Mn,Fe)S present in the form of single precipitates or clusters. Calcium introduced durring the secondary metallurgy changes the morphology of inclusions to a globular form [10]. The formation of spherical CaS inclusion instead of MnS by Ca addition is effective to improve the resistance of steel [11]. The morphology of these inclusions, the presence and morphology of MnS - in particular, significantly affects the steel toughness at low temperatures. In API steels,

attention should be focused on possible occurrence of edge defects in the hot rolled strips. Therefore, the most important task is to cast the slab free from any surface defects, such as various cracks extending from the casting surface to its interior [12,13]. Absence of such defects in the strip is highly desirable and allows for its further trouble-free processing.

2. Materials and methods of investigation

The investigated steel was produced by ArcelorMittal Poland in Krakow, using a continuous casting process. Chemical composition of the test material is shown in Table 1.

To reveal the surface and subsurface cracks in the side part of the slab, non-destructive tests were carried out, including the use of an EPOCH 600 ultrasonic flaw detector provided with the SEB4S, TMAPF 60-4, MWB 45-2 probes, magnetic test (MT) using MPS-F fluorescent magnetic powder and contrast agent - Opti-Lux OLX-365 flashlight, and finally, the flaw detection technique based on the use of an electromagnetic yoke featuring a 160 mm distance between the poles and a field strength of 3200 A/m.

Metallographic examinations of the steel structure carried out in slab corners and middle part of the axis were based on the use of a Neophot 32 light microscope and a Hitachi S-3400N scanning electron microscope (SEM). The X-ray spot analysis and surface analysis of the chemical composition

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Chemical composition of the investigated steel grade API 5L

Chemical analysis	С	Mn	Si	Р	S	Nb	Ti	Al	
	wt. %								
by API 5L [1]	max	max	max	max	max	max	max	-	
	0.16	1.6	0.45	0.025	0.02	0.5	0.04		
analysis from the mould	0.074	1.10	0.21	0.012	0.004	0.028	0.032	0.037%Al	
analysis from the slabs*	0.060	1.08	0.17	0.010	0.006	0.034	0.034	0.030%Al	

*/ carbon equivalent Ceq= 0.243

of phases present in the test steel were carried out with a Noran EDS X-ray microanalysis system.

Hardness was measured on the surface and in the axis of the slab using a portable EQUOTIP durometer equipped with a 5 mm diameter spherical indenter and characterized by an impact energy of 90 N/mm.

The content of non-metallic inclusions was determined on a grid with 300 measuring nodes. Studies were conducted in 20 measurement areas. For the determination of carbon, sulphur, oxygen and nitrogen content, a LECO apparatus was used (e.g. RO16, TN14).

3. Results and discussion

Non-destructive testing (ultrasonic, magnetic particle and penetrant) was carried out on a 400x220x95 mm test piece taken from the slab and subjected before testing to mechanical cleaning (Fig. 1a). The conducted tests and studies showed absence of surface and subsurface cracks on the slab side surfaces and in the corners. Then, specimens measuring 50x50x220 mm were cut out from the slab side edge for further studies (Fig. 1b).

Metallographic examinations carried out in selected areas (dark patches in Fig. 1b) confirmed absence of micro cracks in the microstructure of the examined steel on the side surface, on the transverse surface and in the slab axis. The results have also confirmed that the optimal selection of parameters of the continuous casting process and chemical composition of the manufactured steel, including the amount of the introduced alloying elements, is very important for the presence or absence of cracks.



Fig. 1. View of the slab test piece after mechanical cleaning - a) and schematic picture of the specimen cut out from the slab for further metallographic studies - b)

Hardness was measured on the slab side surface of 220 mm height. The measurements have revealed some minor differences occurring in the hardness values between the slab axis (130÷135 HB) and subsurface areas (120÷123 HB), due to differences in microstructure and process of elemental segregation. Studies using, among others, SEM and LECO apparatus have confirmed slightly different chemical composition in the slab selvedge layer as compared to the middle part of its height (Table 2).

TABLE 2 Chemical composition of the investigated steel in the corner and axis of slabs

	Element, wt. %							
Area	Mn	Al	S*	C*	O*	N*		
Corner of slab	1.039	0.042	0.0038	0.054	0.0056	0.0059		
Axis of slab	1.060	0.039	0.0040	0.059	0.0055	0.0057		
* / chemical analysis made by LECO apparatus: total content of Al and O								

The analysis of the described areas also included gas content (nitrogen and oxygen). The results showed comparable gas content in the selvedge layer and in the axis. Microstructure analysis of the tested steel has revealed the heterogeneity of phase constituents, i.e. ferrite and pearlite, in the tested slab. Figure 2 shows the microstructure of investigated steel was formed by a ferrite phase matrix and pearlite colonies with a relative proportion of each constituent of about 89.3% in the corner and 93.3% ferrite in the axis slabs and 10.7 and 6.7% pearlite respectively.

Studies of non-metallic inclusions in the corners and axis of the examined slab have revealed the presence of complex non-metallic inclusions of a globular morphology (Fig. 3). Microanalysis of the chemical composition showed that part of the area containing precipitates was enriched with Al and O, while in the remaining area, high levels of S and Fe were observed. In the precipitates of both oxides and sulphides, the presence of Ca was detected, said element being used in the secondary metallurgy of steel (Fig. 4, 5). So, the complex precipitates present in the tested steel were globular oxysulphides of a diameter not exceeding $5\div10 \ \mu m$.

Examples of EDS spectra of points selected in Figure 3 is given in figures 4 and 5.



Fig. 2. Microstructure of steel optical microscope a), b), SEM images of steel c),d), scanning electron microscopy, Nital etched;



Fig. 3. Examples of complex non-metallic inclusions occurring in the corner and axis of the examined slab; SE image



Fig. 4. X-ray spectrum with the energy dispersion (EDS) - Fig. 3a



Fig. 5. X-ray spectrum with the energy dispersion (EDS) – Fig. 3b

carried out on the SE and BSE images has revealed that the regions identified as oxides contained up to 24% Al, $43 \div 54\%$ O and up to 25% Ca which would confirm the presence of the 2CaO·6Al₂O₃ inclusion [4]. The presence of iron in an amount of up to 4% Fe was also detected in these areas. The examined steel was some oxides additionally Mg was identified, probably originating from the insulatingrefining powder used in the Continuous Casting process [14].

Areas showing up to 34% S, 15% Fe, 1% Mn and up to 50% Ca confirm the presence of complex sulphides containing, like oxides, high levels of Ca. It is characteristic that manganese was not always found in the areas identified as sulphides (Fig. 4a, 5b, 6) or contained in much smaller quantities than in cast steel [15].

Spot analysis of the oxide/sulphide interface areas showed that they contained $4\div20\%$ S, $2\div14\%$ Al, $15\div25\%$ O and up to 2.4% Mn. The content of Ca in these areas varied within fairly wide limits (Table 3).

Identification of inclusions in the examined material revealed the presence of complex precipitates of oxysulphides, but absence of nitrides. This is probably due to the formation of fine-dispersed precipitates of carbonitrides (e.g. (Ti,Nb)(C,N)) [16] and nitrides introduced to the examined steel with the micro additions of Nb and Ti. Identification of these phases requires the use of transmission electron microscope operating in a STEM mode.

The analysis of non-metallic inclusions in the examined material using light microscopy showed their even distribution in the area of the slab corners and axis. The measurements of non-metallic inclusions in these areas, i.e. on the surfaces longitudinal and transverse to the direction of casting, have indicated a nearly twice as high content of the inclusions in corner areas compared to the axis.



Fig. 6. SEM image of a non-metallic inclusion a) and surface distribution of Fe, Al, S, Ca, Mn in the inclusion - $b \div f$)

TABLE 3

Chemical composition of typical complex inclusions from Fig 3, determined by the X-ray microanalysis method

Point	Element, wt.%								
	S	Al	0	Fe	Ca	Mg	Si	Mn	
1, Fig.3a	3.5	22.1	43.2	4.0	25.1	1.8	0.3	-	
2, Fig.3a	34.4	5.1	-	12.3	47,5	0.3	0.4	-	
3, Fig.3a	32.5	1.1	-	14.9	51,5	-	0.2	0.2	
4, Fig.3a	20.1	11.4	24.0	6.5	36.4	0.9	0.2	0.7	
1, Fig.3b	4.21	13.9	27.8	49.7	3.4	-	-	1.0	
2, Fig.3b	23.2	2.5	15.6	30.6	25.7	-	-	2.4	

4. Conclusions

- 1. The conducted non-destructive testing and inspection of the microstructure of the slab cast from micro alloyed low-carbon steel did not reveal the presence of cracks and micro-cracks in the surface and subsurface layers,
- 2. In the slab corner areas and axis, the presence of nonmetallic inclusions, in prevailing part of a globular morphology, composed mainly of oxysulphides enriched with Ca, has been identified. Absence of single MnS inclusions in steel fully justifies the use of Ca in secondary metallurgy,
- 3. It was found that the content of non-metallic inclusions in the corners and on the surface is nearly two times higher than in the axis,
- Lowering the oxygen content to approx. 55 ppm in the continuously cast steel produced complex non-metallic inclusions of oxysulphides with a diameter of up to 5μm,
- 5. Small differences were found to exist between the hardness of the slab surface and its axis due to differences in microstructure and elemental segregation.

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