IOSUD – Polytechnic University of Timisoara
Doctoral School of Engineering Studies

RESEARCH ON IMPROVING THE STEEL DEGASSING AND DEOXIDATION PROCESSES FOR PIPES

Doctoral thesis – Abstract

for obtaining the scientific title of doctor at

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in the field of PhD Materials Engineering

author eng. MAGAON MIRUNA

scientific coordonator Prof.dr.eng. HEPUȚ TEODOR

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SUMMARY

PREFACE

The metallurgical industry (the steel industry, non-ferrous metals and alloys, metal powders, composite materials, shape memory alloys, special metal alloys, etc.) represents the basis of advanced industrial development for any country. Historically, the steel industry has played an essential role in the creation of the EU, through the establishment of the European Coal and Steel Community in 1951, where European countries developed a framework for long-term economic and political cooperation, with the aim of creating interdependence in key areas in order to avoid the reviving of the war industry, thus ensuring lasting peace on the European continent.

The meaning/explanation behind the difficult situation of the European steel industry is the fact that it has "moved too close to the break-even point", in the area of economic losses, if the EU delays in actions for the overall economic recovery [1/1].

Europe's steel industry is a world leader in certain segments on the steel market, accounting for 1.3% of EU GDP and, in 2015, was providing approximately 328,000 jobs, being at the same time an important source of indirect jobs [2/4].

On international level, but especially on national level, the steel industry is dealing with difficulties in remaining competitive with other economic sectors and, at the same time, with the need to continue to meet the increasingly stringent requirements of steel consumers.

It should be mentioned that after 1990, following the restructuring and the privatization of the national steel industry, the 4 Siemens-Martin steel work factories (of which 3 with integrated flow - 2 in Hunedoara and 1 in Resita - and on non-integrated flow - the Oţelu Roșu steel industry), as well as the electric steel factory no. 1 from Hunedoara with steel casting in the form of ingots were completely decommissioned. Currently, the remaining operating steel work factories (Hunedoara, Reșița, Oțelu Roșu, Târgoviște and Călărași) are equipped with electric arc furnaces type EBT, pot processing installations (LF and VD) and with continuous casting machines (MTC). In Galați there is also an integrated steel work factory (out of 3) with LD type converters and continuous casting (MTC). It is relevant tomention that after restructuring, out of the 6 blast furnaces in operation, only one blast furnace remained, and the cocso-chemical factory is decommissioned (could be considered partially integrated flow).

It is important to note that privatization was aimed at grouping the elaboration flow in the same venture (elaboration-continuous casting-rolling to semi-finished phase) with the rolling flow from semi-finished to finished laminate: in this sense TMK (Resita-Slatina), ArcelorMittal (Hunedoara-Roman) and Tenaris (Călărași-Zalău) are representative.

On the occasion of finalizing my doctoral thesis, I would like to thank all those who guided, supported and shared their professional knowledge with me.

CHAPTER 1- PLAN OF EXPERIMENTS AND RESEARCH

The topic of the doctoral thesis refers to the realization of some studies and researches regarding the elaboration of steels in furnaces with electric arc type EBT, processing in the casting pot (LF and VD processes) and continuous casting. On the elaboration flow, the research particularly focused on the elimination of gases (hydrogen, nitrogen and oxygen) from the steel bath during vacuum processing, purity in non-metallic inclusions, the final goal being to improve the quality of continuous castings and obviously increase the metal removal. The doctoral thesis is structured on 9 chapters, contained in 3 parts.

The industrial experiments took place at TMK S.A. Resita (former Resita Steel Plant), a company specialized in the production of [3/5]:

- round billets Ø 177 mm, Ø 220 mm, Ø 280 mm and Ø 350 mm, continuously cast for alloy and low-alloy carbon steel pipes, for rolling mechanical pipes, heat exchanger pipes, oil / gas pipes , pressure pipes and construction pipes;
- round semi-finished products Ø 177 mm, Ø 220 mm, Ø 280 mm and Ø 350 mm, continuously cast from carbon steel, low alloyed and alloyed, intended for hot plastic processing by forging / molding (flanges, rings, axis, sprockets);

TMK S.A. Resita has modern aggregates, installations and equipment for the development of high quality products, namely: electric arc furnace type EBT (Excentric BottomTapping) of 100 t, pot oven (LF-LadleFurnace) of 100 t, liquid steel vacuum type installation RV (Vacuum-Degassing) capacity 100 t; Continuous Casting Machine (MTC) with 3 curved wires. The maintenance of the production equipment is performed by its own maintenance and repair department.

The applied research strategy (according to the model in fig.1 / 1.1.) used for the elaboration of the doctoral thesis consisted in completing the following stages:

- bibliographic study from the specialized literature regarding:
- elaboration of steel in electric arc furnaces type EBT, processed in the casting pot and continuously cast;
- reduction of the gas content (H, N and O) in the steel bath;
- techniques for analyzing non-metallic inclusions in steel
- research and industrial experiments on establishing correlations between the parameters of vacuum steel processing and continuous casting on:
- the final hydrogen content of the cast steel and the hydrogen removal efficiency, the hydrogen uptake on the VD → MTC technological route;
- the final nitrogen content of the cast steel and the nitrogen removal efficiency, the amount of nitrogen removed, the nitrogen uptake on the technological route VD → MTC;
- final oxygen content of vacuum steel and analysis of non-metallic inclusions;
- industrial verification of the research results performed;
- final conclusions, original contributions, dissemination of research results.

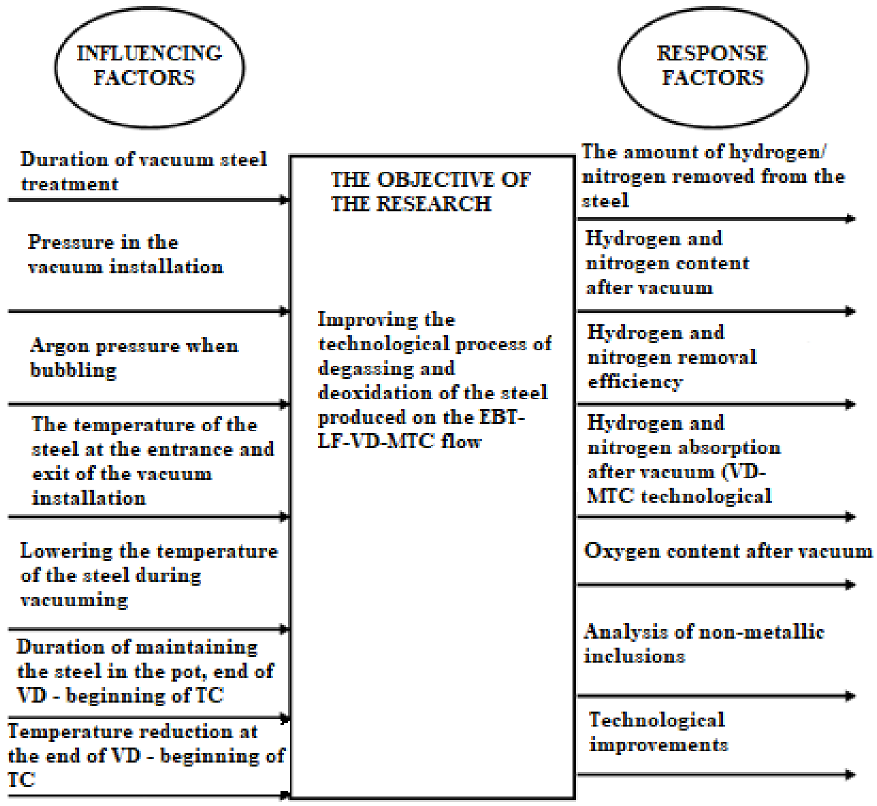


Fig. 1/(1.1.) The input-output model of the research object

PART 1-STUDY FROM THE SPECIALTY LITERATURE REGARDING THE ELABORATION IN ELECTRIC ARC FURNACES AND THE CONTINUOUS CASTING OF STEEL

CHAPTER 2 - EBT TYPE OVEN MANUFACTURING, POT PROCESSING AND CONTINUOUS CASTING OF STEEL

The steel (alloy of iron with carbon) is one of the most important materials of our time, the active part of the fixed funds of the economy being produced from steel.

Currently worldwide, most electric steel works factories are equipped with E.B.T. high power electric arc furnaces, pot processing plants and continuous casting machines.

The electric arc furnace, as a result of the improvements brought both from constructive and technological points of view, as well as its coupling in the technological flow of elaboration with pot treatment installations, allowed, on one hand, the development of "Pot Metallurgy" , and, on the other hand, the extension of the continuous casting process to electric steel works factories as well.

Equipping electric steelworks with EBT type electric arc furnaces and pot processing installations (without or with heat input) LF, RH, VD, VAD, VOD, etc., ensures the development of both ordinary carbon steels or low-alloy steels, as well as of the high allies, they are built within very wide capacity limits [4/15, 5/18, 6/30].

The stages of producing steel in the electric arc furnace type EBT are: ?loading of the electric arc furnace with scrap metal, heating the load, melting; refining and evacuation, pouring into the casting pot (fig.2 / (2.3.)).

The addition to the steelmaking process of steel processing technologies in the casting pot, initially in installations without heat input (inert gas bubbling, inert gas bubbling + vacuuming) followed by those with heat input, led to their grouping under the name of "**POT METALLURGY**", frequently known also as "**SECONDARY METALLURGY**" [4/15, 7/33]. Image 3 (2.5.) presents the sketch of the technological process of treatment in the casting pot [4/15, 7/33].

The steel processing in the LF plant ensures not onlyobtaining improved metallurgical effects, but alsothe synchronization of the batches at continuous casting (of particular importance when casting sequential), and the RV vacuum installation has the role of degassing the steel [4 / 15,7 / 33].

The charge time for EBT electric arc furnaces is between 40 - 80 min (very close to that with oxygen converters), and the productivity is on average 100t / h;

LF installations operate in a duplex system with the primary processing unit, the electric arc furnace or the oxygen converter, these can be considered in the current stage as "buffer stations" between the steel works plants and the continuous casting machines (MTC).

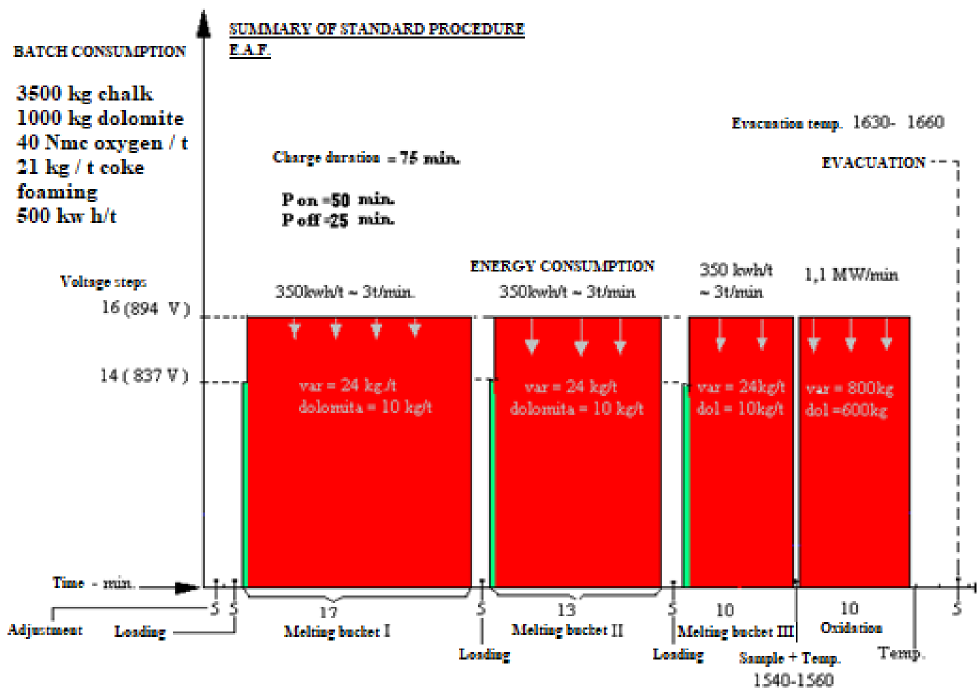


Fig. 2/(2.3.) Diagram of the technological process of elaboration [15].

With the improvement of processing technologies, it was natural to further improve the casting technologies, specifically a transition took place from casting in the form of ingots to continuous casting melt by melt, respectively sequential.

The extremely fast progress of continuous steel casting, both from a metallurgical point of view and in terms of plant construction, has led to the intense development of this process in the steel industry [8/34], due to the following advantages: removal of metal from 82-85% for conventional casting to min. 99% for sequential continuous casting, reduction of investments, energy consumption, reduction of labor, etc.

Currently worldwide and nationally, sequential casting is the standard technology used for all continuous casting installations (but the number of batches cast in a sequence and the frequency of sequences differs), which are equipped with devices for rapid change of the casting pot and distributor, the current trend being to pour semi-finished products with a section as close as possible to the final one.

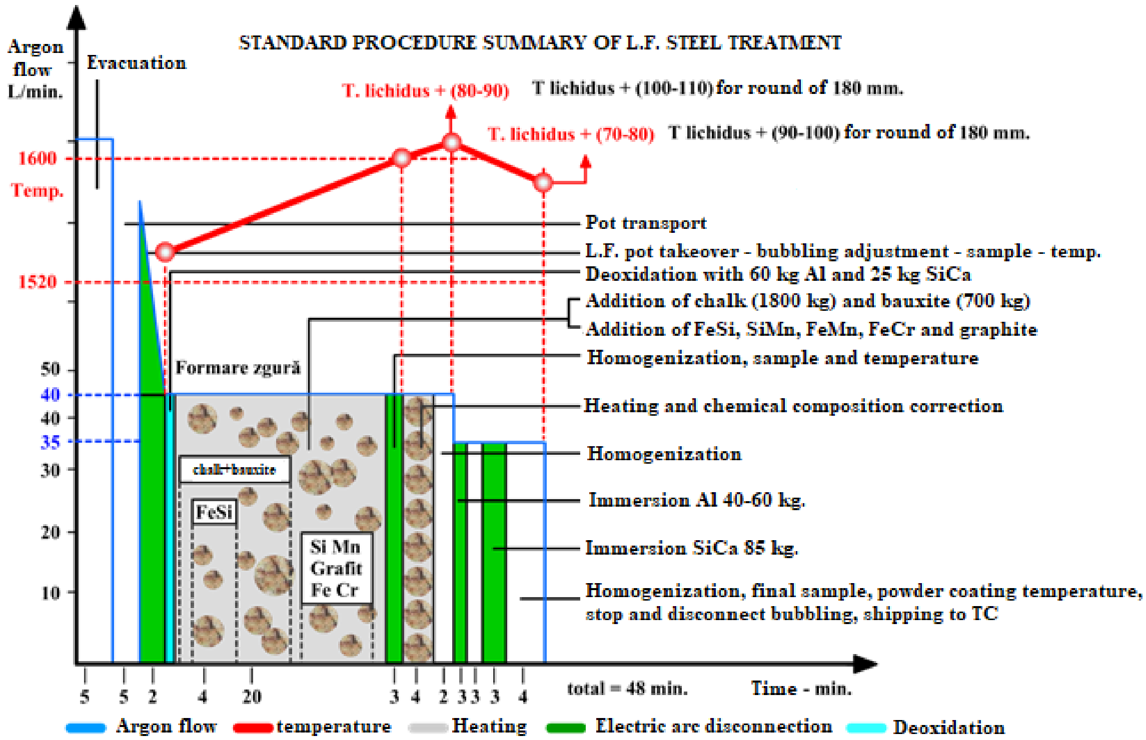


Fig. 3 (2.5.) Diagram of the technological process of treatment in the casting pot (LF)) [15]:

CHAPTER 3 - STUDY FROM THE SPECIALTY LITERATURE ON REDUCING GAS IN THE STEEL BATH

The removal of gases from the steel bath is a permanent problem for metallurgical engineers, which arose with the development of steel production, so this technological process is increasingly being studied in recent years, especially in the refining operation.

The minimization of gas content (H, N) as a major objective in sustainable steel manufacturing programs must be correlated with the knowledge of the influence of gases on steel quality, estimated throughthe values of various quality characteristics, and their behavior in operation.

Hydrogen has a negative influence on the quality of steel [4/15, 9/32], because: it is one of the causes of the appearance of sulphide in ingots and cast parts of calmed steels; it contributes to the appearance of the defect called "flakes" (very small star-shaped cracks) in steels alloyed with chromium and nickel, which substantially reduce the fatigue strength of steel parts; it reduces the plasticity and toughness of steel; it affects the electrical and magnetic properties of steels.

The negative influence of nitrogen in steels manifests as follows [4/15, 9/32]: it reduces the plasticity and toughness of steel, in association with hydrogen, it contributes to the appearance of sulphide in cast steel; it causes the phenomenon of aging of steel by nitride deposition at the limits of structural grains.

From the study of the specialized literature [10/16, 11/25], the following can be considered as main sources of hydrogen and nitrogen in electric steelworks plants (electric arc furnaces type EBT): the metal load itself (assortments of ferrous waste, cast iron, ferroalloys, iron sponge, metallized pellets, metallized chipboard, etc.); moisture of the metal load, it is not always technically possible to preheat the load from the bucket, the additions necessary for the formation of slag, ore, limestone, lime, topex, etc., as well as the supply of lime and dolomitic lime, during transport and parking in the bunkers storage absorbs moisture from the atmospheric air (especially where it does not have its own lime factory), depending on the duration of residence, granulation, degree of combustion; the atmosphere of the furnace, due to the constructive methods (panels, vault and other water-cooled elements) there may occur accidental cases of breaking these elements and for short periods of time, but with high working pressures, the cooling water enters the atmosphere of elaboration the ionizing effect of the atmosphere of the furnace generated by the electric arc, which contributes to the dissolution of N and H especially in the case of a very thin layer of slag.

It should be noted that the solubility of gases in the steel bath is influenced by the accompanying elements and especially by the allied elements.

In general, hydrogen and nitrogen pass through the gaseous atmosphere of the steel processing unit not directly, but through slag (the solubility being lower acid slag).

Favorable conditions that ensure a minimum content of hydrogen and nitrogen in steel are: the use of raw materials with low moisture content or H and N (metal filler, ore, lime, limestone, dolomitic lime, ferroalloys, etc.); ensuring a sufficiently high decarburization speed, more precisely higher than the critical ones (vc> vccr); after deoxidation vc = 0 and therefore V.elimH, N = 0; bubbling with an inert gas (usually argon), and in the case of stainless steel alloyed even with nitrogen; desirable vacuum in installations with heat input.

The degree of degassing generally varies greatly depending on the solubility of the gases in the steel, their condition (free or in combination) and certain characteristics of the vacuum treatment processes, the vacuum parameters, as benchmarks it can be admitted that it is removed 60 - 70% of hydrogen and 0 - 40% of nitrogen (on average 30%) [9/32, 11/25].

CHAPTER 4 - OXYGEN REDUCTION IN STEEL BATH STUDY

In the process of steelmaking, it is known that, with the decrease of the carbon content in the steel, the oxygen content increases, which makes that at the end of the refining period the dissolved oxygen content is relatively high (0.02 - 0.06%, respectively 200 - 600 ppm) and increases strongly when [C] falls below 0.20%.%, Such contents not being allowed in steels because it damages the quality by: “hot embrittlement” which manifests itself in increased susceptibility to overheating and the appearance of cracks in hot deformation time due to oxide and oxisulfide deposits at the structural grain boundary; the formation of blows (metal voids) during the solidification of the cast product (specific for non-quenched and semi-alloy cast steels in the form of ingots - insignificant in the current stage);

For the reasons mentioned, the dissolved oxygen content in the calm steel must not exceed the limits of 0.0040 - 0.0080% (40 - 80ppm), and 150 - 300ppm in the semi-calm and non-calm steel. Deoxidation is done by the following methods: precipitation, extraction / diffusion and with the help of vacuum.

Deoxidation by precipitation consists essentially in the introduction into liquid steel of some materials, **called deoxidizers**, which have in their composition elements with higher affinity for oxygen than iron, this being **a first condition for an element to be deoxidizing.**

The deoxidizing element combines with the dissolved oxygen in the steel and forms stable and insoluble oxides in the liquid steel, which separates (precipitates) in distinct oxide phases, this being **the second condition for the element to be deoxidizing**.

The deoxidizer must have high specific gravity and granulation to be able to penetrate liquid steel (or at least at the slag-steel interface) and react with oxygen dissolved in steel, one of the reasons why in the form of ferroalloys. Another technological solution is to immerse the ferroalloy in the steel bath in the form of wire (aluminum wire) or filled wire (silicon-calcium, aluminum-calcium-silicon). As a result, these aspects **are a third condition for an element to be deoxidizing.**

In order for an element to be used in practice as a deoxidizer it must be economically convenient, **so this is practically the fourth condition imposed for a deoxidizing element**.

In practice, the usual simple deoxidizers (FeMn, FeSi, Al) are used, as well as complex (SiMn, SiCa, AlCaSi), and special (FeTi, FeV, FeNb, FeB, PR). [10/16, 11/25].

Deoxidation of steel by extraction / diffusion is based on the fact that oxygen, respectively ferrous oxide FeO, is soluble in both steel and slag, and as a result of its deoxidation, [FeO] → (FeO), the steel deoxidizes. This method can be applied to the elaboration of steel in the electric arc furnace or in the casting pot (to any elaboration unit).

Vacuum deoxidation is one of the most efficient processes for deoxidizing steel with multiple applications in the production of high purity steels.

The process of removing non-metallic inclusions from liquid steel takes place largely simultaneously with the process of their formation and growth, of particular importance being the steel-inclusion interphase voltage, their specific weight, the increase in any way of the speed of removal of inclusions (bubbling , vacuuming, remelting, etc.), [10/16, 11/25].

It should be mentioned that, during bubbling with argon / nitrogen, the elimination of unmetallic dry inclusions of steel can also take place, through the flotation phenomenon [10/16, 11/25].

The destruction / reduction of non-metallic inclusions in vacuum is based on the fact that the affinity of carbon for oxygen increases so that it is a very strong deoxidizer, reduces the inclusion and CO leaves the metal bath [10/16, 11/25].

PART II

RESEARCH AND EXPERIMENTS REGARDING DEGASATION AND DEOXIDATION OF STEEL PROCESSED IN VD TYPE DRAINAGE INSTALLATIONS.

CHAPTER 5 - RESEARCH AND EXPERIMENTS REGARDING THE REDUCTION OF HYDROGENAL CONTENT STEEL PROCESSING IN VD TYPE-INSTALLATIONS

The research conducted and the obtained results presented in this chapter focused on the possibilites to reduce the hydrogen and to increase the efficiency of its removal in the steels designed for pipes manufacturing.

The researches were carried out at an electric steelworks plant, equipped with an electric arc furnace type EBT, LF installation, RV installation and 3-wire continuous casting installation. On the technological flow presented (Chapter I), a number of 22 steel batches brand ST 52-3 continuously cast in round billet of ϕ280mm were analyzed. These researches aimed to establish technological correlations between dependent and independent parameters, as follows:

- **dependent parameters**: hydrogen content in the bath at the end of the vacuum [H] fVD, ppm, hydrogen removal efficiency RH,%.
- **independent parameters**: total vacuum duration (Dtv), min; the duration of ultra-high vacuuming (Dvid.av) min; the pressure in the vacuum facility under ultra-high vacuuming (Pvid.av), mBar; the bubbling argon pressure (PbAr) in VD, atm, the temperature reduction during vacuum (ΔT), ^{0}C; the temperature of the steel at the entrance to the vacuum system (TinVD), ^{0}C; the temperature of the steel at the exit of the vacuum system (TisVD), ^{0}C;

Once the values for the mentioned parameters were known, the data was processed in the EXCEL and MATLAB calculation programs.

Within the EXCEL calculation program, polynomial correlation equations of degree 1; 2; 3; 4 and logarithmic were established, in each case the results being presented graphically and analytically. Also for each case the correlation coefficient is presented.

By processing the data in the MATLAB calculation program, multiple (double) equations resulted between the dependent parameter (z), the independent ones (x and y) and the a_1-a_9 coefficients, the results being presented both analytically and graphically (the surface of correlation, level curves spatial and plane projection).

To estabilish the correlations, the following functions / equations were used:

Equation 1 z = a1 + a2x + a3x2 + a4x3 + a5y + a6y2 + a7y3 + a8y4 + a9y5 ((1 / (5.2))

Equation 2 z = a1 + a2logx + a3 (logx) 2 + a4 (logx) 3 + a5 / y + a6 / y2 + a7 / y3 + a8 / y4 + a9 / y5 ((2 / (5.3))

Equation 3 z = a1x2 + a2y2 + a3xy + a4x + a5y + a6 ((3 / (5.4))

The following are examples of simple and multiple correlations (expressed analytically and graphically), as well as the technological analysis of these correlations [12/58, 13 / 61,14 / 64,15 / 68]

The increase of the total vacuum duration of the steel in the VD type vacuum installation leads to the advanced reduction of the final hydrogen content (fig. 5.1.). It is desirable that the total vacuum duration be between 25-28 min., which is very good for a vacuum installation without heat input during the treatment, thus ensuring a hydrogen content of 0.8-1.1ppm.

Correlation 1: [H] fVD = f (Dtv, Dvid.av) shown in Figures 5.13; 5.14 and 5.15 with spatial coordinate points A (30; 19; 0.9); B (30; 19; 1.0) and C (30; 19; 1.1), respectively. A difference of 0.2ppm [H] fVD is permissible given max. of 1.1ppm. Very good results are obtained for Dtv = 25-31 min and Dvid.av = 15-20 min, resulting in [H] fVD below 1.4ppm; Similarly, the other graphical representations in this chapter are analyzed.

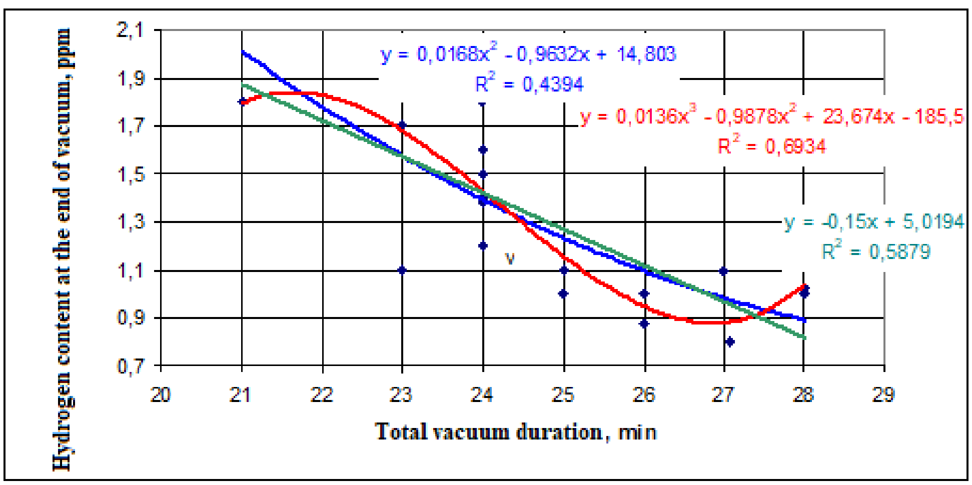
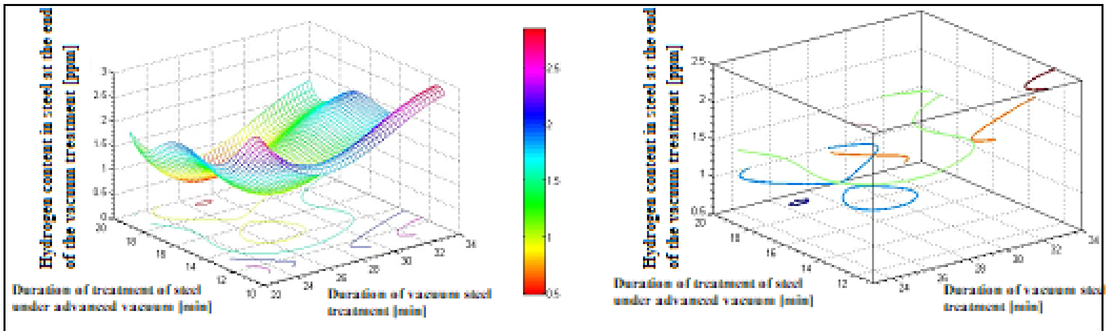
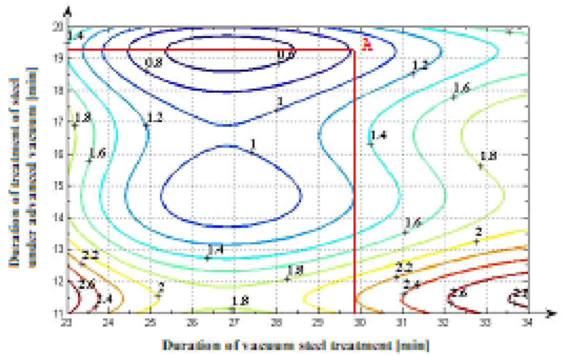


Fig4/(5.1.) Correlation $[H]_{fVD} = f(D_{tv})$



a) Correlation surface

c) Contours, projection in space



b) Contours, projection in plan

Equation type 1

$D = 50143.37411$; $H = -2291.32815$;
$R^2 = 0.69283$; $r^2 = 0.68779$
$a_1 = -1145.66407$; $a_2 = -10.94201$;
$a_3 = 0.361301$; $a_4 = -0.003909$;
$a_5 = 426.291125$; $a_6 = -57.08538$;
$a_7 = 3.77343$; $a_8 = -0.12323$;
$a_9 = 0.00159$

Fig.5 (5.13.) Correlation $[H]_{fVD} = f(D_t$ $D_{vid.av.})$

d) Correlation Equation

CHAPTER 6

RESEARCH ON CONTENT REDUCTION OF NITROGEN BY VACUUM IN STEEL FOR PIPES

The research whose results are presented in this chapter took place in the same experimental technological framework as those presented in Chapter 5, with the following clarifications:

- the independent parameters are the same;
- the dependent parameters are: the nitrogen content at the end of the vacuum; nitrogen removal efficiency during vacuuming, the amount of nitrogen removed during vacuuming.
- the data were processed in the EXCEL and MATLAB programs [16 / 65,17 / 63].

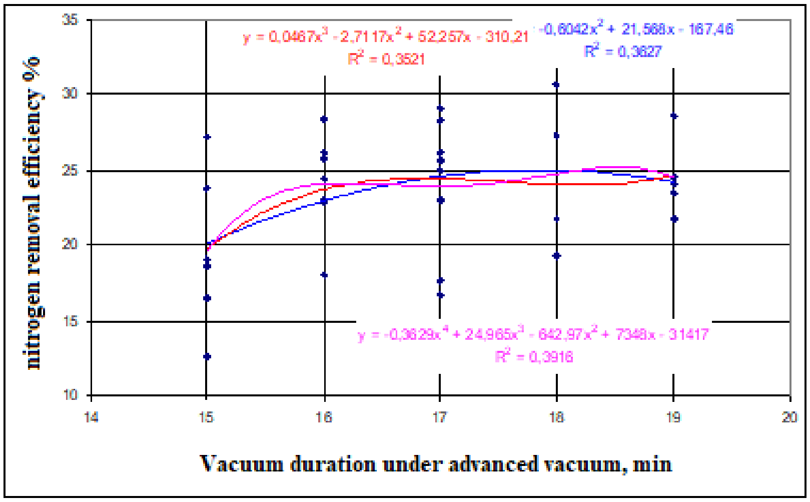
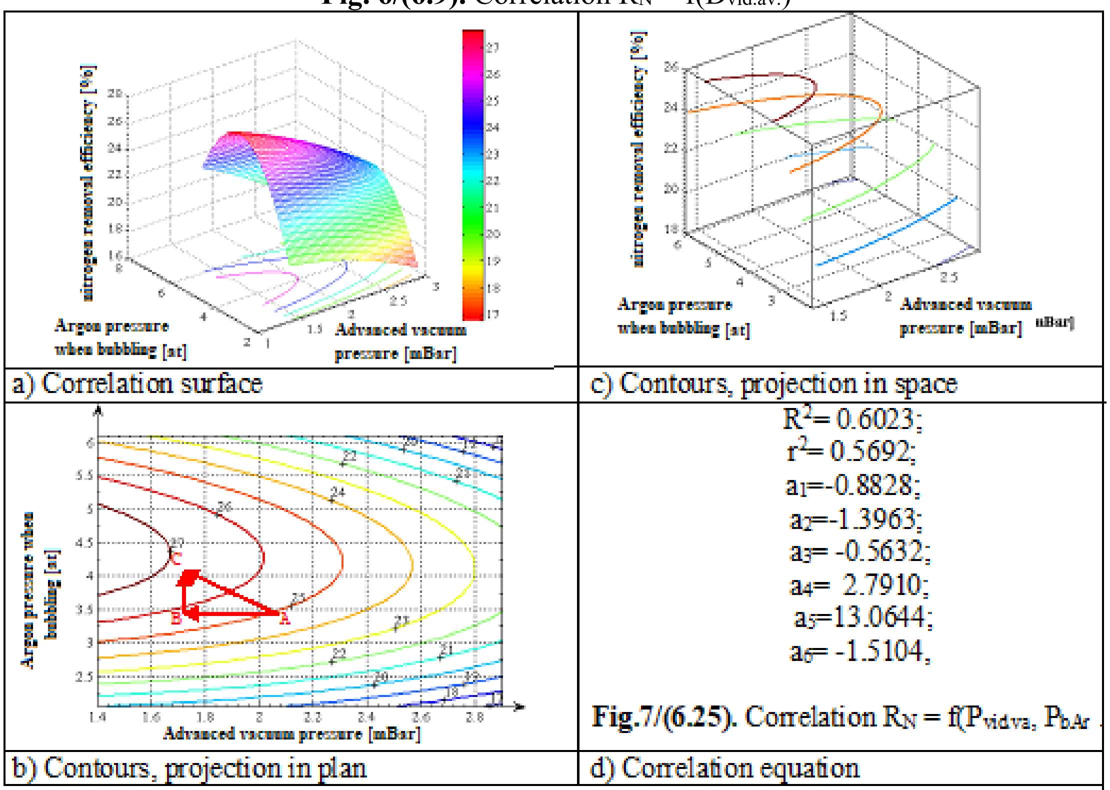


Fig. 6/(6.9). Correlation $R_N = f(D_{vid.av.})$



$R^2 = 0.6023$;
$r^2 = 0.5692$;
$a_1 = -0.8828$;
$a_2 = -1.3963$;
$a_3 = -0.5632$;
$a_4 = 2.7910$;
$a_5 = 13.0644$;
$a_6 = -1.5104$,

Fig.7/(6.25). Correlation $R_N = f(P_{vid va}, P_{bAr})$

d) Correlation equation

By processing the data in the EXCEL program, polynomial correlation equations of degree 1; 2; 3 and 4, were established, in each case the results being presented both graphically and analytically. In the MATLAB program the data was processed in order to obtain double correlations of degree 2 (equation 3, chapter 5). Below is the technological analysis of the 2 correlations presented above.

- regarding the influence on the nitrogen removal efficiency, the duration of vacuum under advanced vacuum (fig.6 / (6.9)), there is an increase of the nitrogen? to values of 25-30%, for Dvid.av = 17-19 min.; the results obtained correlate very well with those from the total duration of vacuum Dtv = 25-29 min, thus being able to lower the nitrogen content at the end of vacuuming (60-70ppm);

- the data presented in fig. (7 / (6.25)) shows the influence of independent technological parameters, the pressure in the installation under advanced vacuum and the argon pressure for bubbling steel during vacuuming on the dependent parameter, the nitrogen removal efficiency from the steel bath. The increase of the argon pressure when bubbling and the decrease of the pressure in the vacuum installation positively influences the nitrogen elimination efficiency because it generates favorable conditions for degassing, (Ex. A (15; 3.5; 24) → B (16; 3.5; 26) → C (16; 4.8; 28)).

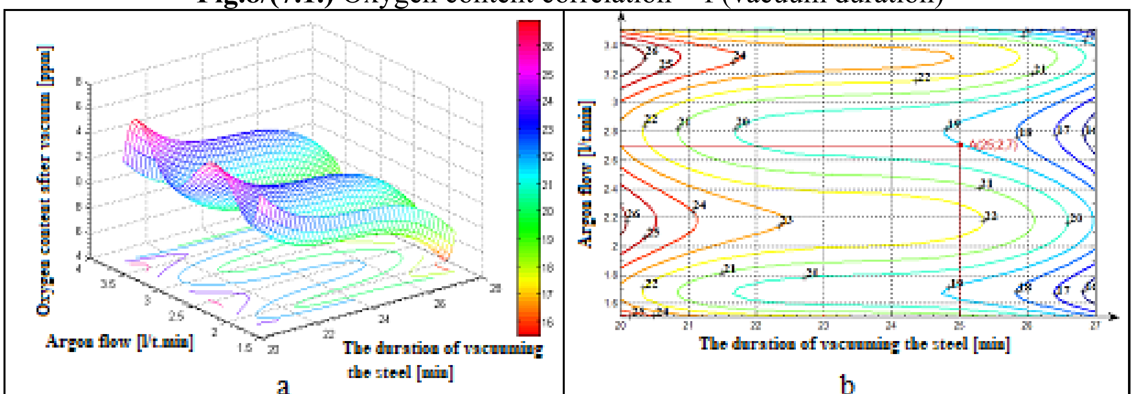
The thesis also presents triple correlations, both in analytical and graphical form. The process of nitrogen absorption on the VD-TC technological route is also analyzed.

CHAPTER 7
RESEARCH ON REDUCING OXYGEN CONTENT BY IN STEEL FOR PIPES

The theoretical basis for reducing the oxygen content of steels [18/48, 19/57] is presented in Chapter 4, consequently this chapter presents a series of results obtained in industrial experiments carried out in the same technological framework as those presented in chapters 5 and 6. Regarding the research carried out, only one dependent parameter was chosen - the oxygen content of the steel after vacuum -, the independent parameters being the same as in the other two cases (hydrogen and nitrogen).

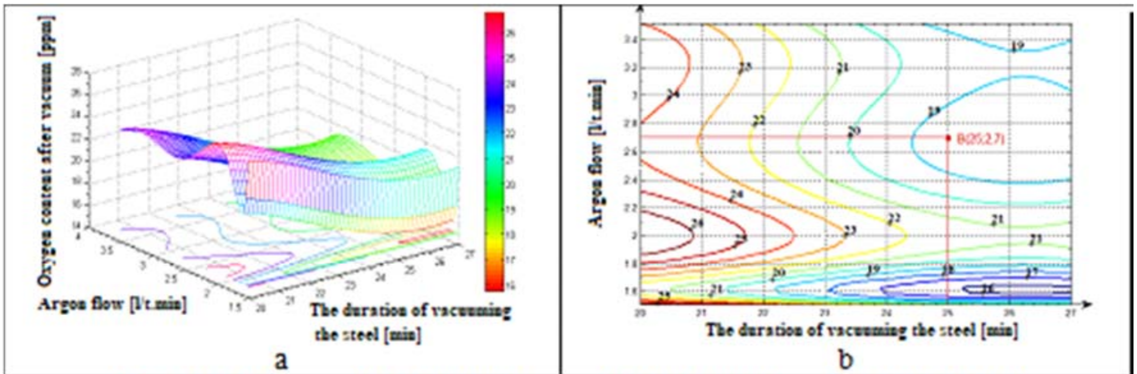
Also in this case, the technological data was processed in the EXCEL program for single correlations and in MATLAB for double correlations, in both cases the results obtained being presented both analytically and graphically (in the first part of this chapter) and accompanied by technological analysis. The following are examples of such correlations.

Fig.8/(7.1.) Oxygen content correlation = f (vacuum duration)



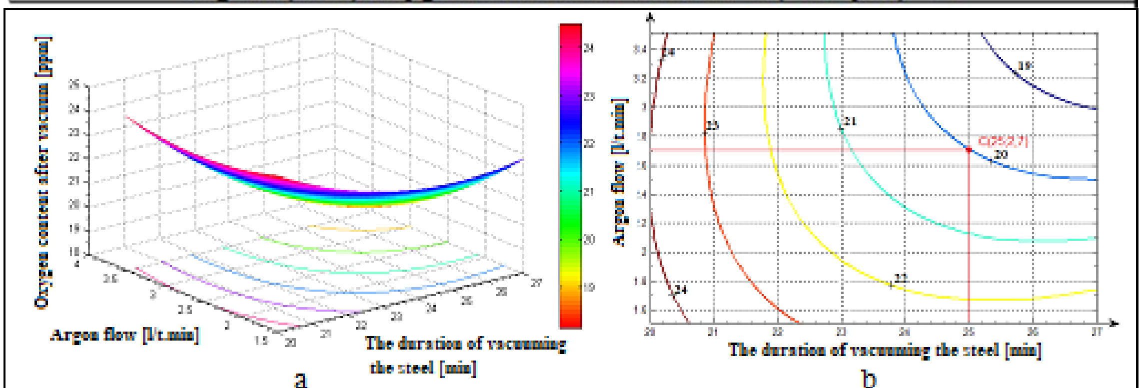
R^2=0.928125; a_1 = 3080.857397; a_2= -121.935601; a_3 = 5.198436; a_4 = -0.073958 a_5 = -4619.131059; a_6 = 3959.522994; a_7 = -1658.466332; a_8= 339.756656; a_9 = -27.267369;

Fig.(9/(7.9.) Oxygen content after vacuum = f(D_{tv}, $Q_{b.Ar.}$) – A



R^2=0.935203; a_1 = -12460.878773; a_2 = 10951.127731; a_3 =- 3531.540514; a_4 = 378.636858; a_5 = 14475.987866; a_6 = -68770.357779; a_7 = 159643.168303; a_8 = -180970.016205; a_9 = 80151.536062;

Fig. 10/(7.17.) Oxygen content after vacuum = f (D_{tv}, Q_{bAr}) – B



R^2 = 0.848899; a_1 = 0.081470; a_2 = 0.454855; a_3 = -0.319134; a_4= -3.598831; a_5 = 4.055380; a_6 = 66.342935;

Fig. 11/(7.25.) Oxygen content after vacuum = f(D_{tv}, Q_{bAr}) – C

Regarding the duration of the treatment of the steel under vacuum (fig.8 / (7.1), it is found that with the increase of these durations, a decrease of the oxygen content is ensured, technologically explainable by longer diffusion time of the oxygen from the steel bath in the vacuumed space. From the analytical presentation of these equations. we find very close values for regression coefficients, and in the graphical representation, they practically coincide. Given these aspects, it can be considered that in the current research and in practice, we can use linear correlation. Similarly, the other simple correlations are being analyzed.

Regarding the correlations, they were established according to the three types of equations (5.2) graph A; (5.3) graph B and (5.4) graph, these being analyzed unitarily and comparatively, as follows: **Correlation 2**: $[O]_{fVD}$ = f(D_{tv}, Q_{bAr}) shown in figures 9 / (7.19), 10 / (17) and 11 /(7.25) with spatial coordinate points A (25; 2.7; 19); B (25; 2.7; 18.75) and C (25; 2.7; 20), respectively. Differences of -1.0 ppm, -1.25 ppm and 0 ppm $[O]_{fVD}$ are permissible given max. of 26 ppm, and even more so that it is below the reference (20 ppm). Very good results are obtained for D_{tv} = 20-28 min and Q_{bAr} = 2,5l / t.min- 3,5l / t.min, being able to obtain $[O]_{fVD}$ below 24ppm;

Part II of this chapter presents a series of results regarding the nature and chemical composition of non-metallic inclusions contained in continuous castings (quality AMJ 21C). The nature of the inclusions was determined by microstructural analysis performed by scanning electron microscopy (SEM) and energy-dispersive X-ray microanalysis (EDAX) on the ***QuantaInspect F.*** scanning electron microscope.

From the study of the analysis of the chemical composition of the inclusions from the studied samples (in total 6) the following can be highlighted:

- Variation limits for the three main (predominant) elements of the analyzed inclusions (microarias with zone 1) in samples 1; 3; 4 and 5 are:

- O = 26.81 - 27.72 in% by weight (average 27.26%);
- O = 43.44 - 44.38 in atomic% (average 43.91%);
- Al = 20.65 - 25.71 in% by weight (average 23.18%);
- Al = 20.06 - 24.59% atomic% (average 22.32%);
- Ca = 41.93 - 42.18 in% by weight (average 42.05%);
- Ca = 26.01 - 27.72 in atomic% (average 26.86%);

- The share of the three cumulated elements (O + Al + Ca) in the composition of the inclusions varies within the limits: 86.41- 95.61%% by weight, respectively 89.51-96.69% atomic (the average values being 91.01% by weight, correspondingly 93.10% atomic; it should be noted that very rarely extreme values can be reached).
- The elements magnesium (Mg), silicon (Si), sulfur (S) and iron (Fe), present together with the other three elements mentioned above in zone 1 of the micro-areas analyzed, have a relatively small share in the composition of the inclusions within the limits: 5, 73-11.82% weight (average 8.78%) and 4.96- 9.49% atomic (average 7.22%).

In the third (last) part of this chapter, series of results are being presented regarding the increase of the microscopic purity of the steels produced at TMK Resita. During the industrial experiments provided in the plan for the elaboration of the doctoral thesis, this steel company used several deoxidation technologies that proved their efficiency, and the following aspects were taken into account:

- establishing the deoxidants used, the specific addition, the state of presentation of the deoxidant, the order of the addition in the oven, pot, Lf installation, respectively VD;
- establishing the treatment parameters in LF and VD;

The technology for the elaboration of fine crystalline grains is being analyzed, specifying the importance of the [Ca] / [Al] ratio: 0.92-1.02 (considered optimal ratio).

During the experiments, the impurity of the steel (quality 4140) with exogenous inclusions (dust from the crystallizer due to the cracking of an immersion tube (tube 1) was also analyzed.

Impurity of cast steel on wire 1, with inclusions about 10 times the size of F2 and F3, occurred when the immersion tube cracked; the flow of liquid steel through tube 1, entrained lubricating dust from the crystallizer, fact confirmed by the composition of the inclusions.

CHAPTER 8
INDUSTRIAL VERIFICATION OF THE RESULTS OBTAINED IN RESEARCH

Technological considerations

The results obtained in the research were verified in practice at the same company, on a number of five steel loads (the same brand as in the experiments and the same technological flow). It should be mentioned that, regarding the structure and quality of the load (raw and auxiliary materials), there were no significant differences (variations in the usual limits), respectively the steel processing in the LD and VD installations took place without any technological deviation.

The technological data obtained during the industrial verification of the research results confirms the validity of the research and the possibility of their application in current practice.

PART III
FINAL CONCLUSIONS AND ORIGINAL CONTRIBUTIONS
CHAPTER 9
FINAL CONCLUSIONS. ORIGINAL CONTRIBUTIONS. DIRECTIONS FOR CONTINUING RESEARCH

Final conclusions

After the restructuring of the steel industry (period 1990-2000), for the production of steel in Romania the following types of aggregates are used: electric arc furnaces (type EBT) and oxygen converters (type LD). The final conclusions refer to the elaboration of steel in electric arc furnaces type EBT, continuous casting, elimination of gases from the steel bath (hydrogen and nitrogen) and deoxidation in vacuum installations without heat input (type VD.). Correlations obtained by data processing in the EXCEL and MATLAB computer programs, are presented in both analytical and graphical form, always accompanied by technological analysis and practical application possibilities.

Original contributions

Based on the study of the literature, relevant for the topic of the doctoral thesis, on the experiments performed at the industrial phase level, the processing of experimental data and technological analysis of the results obtained, as well as their industrial verification, the following can be considered personal / original contributions :

1) the elaboration based on the study of the specialized literature regarding the topic of the doctoral thesis of the syntheses/summaries regarding: the elaboration of steel in electric arc furnaces and continuous casting, the elimination of hydrogen and nitrogen from the steel bath and the deoxidation of steels;

2) establishing by processing experimental (industrial) data in the EXCEL calculation program of some (simple) polynomial correlation equations of degree 1; 2; 3; 4 and logarithmic equations, and in the MATLAB a and double calculation program, respectively, between the main parameters of vacuum considered independent and the hydrogen content of steel after vacuuming and hydrogen removal efficiency, considered dependent, in each case the results being presented both graphically and analytically, as well as technologically analyzed;

3) obtaining the simple and respectively double correlations (in the EXCEL and MATLAB programs) by processing the data regarding the hydrogen absorption in the steel bath on the technological route VD - distributor, followed by the technological analysis.

4) processing experimental (industrial) data in the EXCEL calculation program and obtaining (simple) polynomial correlation equations of degree 1; 2; 3; 4 and logarithmic equations, between the main parameters of the vacuum considered independent and the nitrogen content of the steel after vacuum, the nitrogen removal efficiency and the amount of nitrogen removed, considered dependent, in each case the results being presented both graphically and analytically, as well as analyzed from a technological point of view;

5) analyzing the data regarding the nitrogen removal efficiency, determining and processing it in the MATLAB program, thus obtaining double correlations expressed by polynomial equations of 2 degree (independent parameters the same as for simple correlations), and presented both analytically and graphics, being accompanied by technological analysis;

6) establishing, through processing in the MATLAB program, the data regarding the nitrogen elimination yield of some triple correlations expressed by polynomial equations of 2 degree (independent parameters the same as the simple correlations), and presented both in analytical form, being accompanied by technological analysis ;

7) establishing, by processing in the MATLAB program, the data regarding the amount of nitrogen of triple correlations expressed by polynomial equations of degree 2 (independent

parameters the same as for simple correlations), and presented both analytically and graphically, being accompanied by technological analysis; for the graphical presentation, the triple correlations were transformed into double correlations (a triple correlation generates three double correlations), these representations being accompanied by a technological analysis:
8) processing, in the EXCEL and MATLAB calculation programs, the data regarding the nitrogen absorption in the steel bath on the technological route VD - distributor and obtaining simple and double correlations, respectively, followed by the technological analysis.
9) establishing, by processing experimental (industrial) data in the EXCEL calculation program, some (simple) polynomial correlation equations of degree 1; 2; 3; 4 and logarithmic equations, between the main parameters of the vacuum considered independent and the oxygen content of the steel after vacuum, in each case the results being presented both graphically and analytically, as well as analyzed from a technological point of view;
10) determining, by processing in the MATLAB program, the data regarding the oxygen content in steel after vacuum of some double correlations expressed by polynomial equations of degree 2 (independent parameters same as for simple correlations), of degree 5 and a combined / mixed polynomial form + logarithmic, in all cases being presented both in analytical and graphical form, accompanied by technological analysis;
11) determinining the nature, composition and size of non-metallic inclusions (pipe steels) by microstructural analysis performed by scanning electron microscopy (SEM) and by energy dispersive X-ray microanalysis (EDAX) under the scanning electron microscope ***Quanta Inspect F;***
12) the elaboration of a liquid steel processing technology on the CAE-LF-VD-MTC manufacturing flow which reduces the clogging phenomenon, thus avoiding the clogging of the immersion tubes;
13) Analysis of the characteristics of non-metallic inclusions in the semi-finished product, generated by the cracking of the immersion tube;
14) Industrial verification of the research results performed, the results obtained confirming their validity / importance;
15) Dissemination of research results and directions.

Directions for further research

Based on the results obtained in the research conducted on the topic of the doctoral thesis, it can be considered that it is of particular interest to the metallurgical / steel industry and not only, the continuation of research in the following directions:

1) the absorption of oxygen in the liquid steel after finishing its processing in the vacuum installation (without or with heat input) on the technological route vacuum installation - distributor - crystallizer - semi-finished product;

2) the influence of the casting powders quality (distributor and crystallizer) on the gas absorption in the continuously cast steel semi-finished product;

3) clogging of the immersion tubes depending on the deoxidants used and the quality of the casting powders (ointments).

DISSEMINATION OF RESULTS

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