INCLUSION CONTROL AT ÇEMTAŞ STEEL PLANT

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ABSTRACT

INCLUSION CONTROL AT ÇEMTAŞ STEEL PLANT

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ÇEMTAŞ Steel Plant produces alloy steels used principally by the automotive industry. Demands of customers for cleaner steel with "fewer, smaller and homogenously distributed inclusions" are getting tighter with time. The tight demands of the customers are checked by ultrasonic testing and faulty regions are cut off and scrapped which results in production losses in steel plants. Decreasing production losses due to scrapping by producing cleaner steels is therefore a major concern. The objective of this study is to investigate factors affecting the cleanliness of the steels produced by ÇEMTAŞ. With this objective, the steelmaking practice used was thoroughly investigated and the possible origins of inclusions in the final product was tried to be found. Based on the findings, process parameters like temperature, time, quantities of additives to steel, intensity of stirring etc. were varied with the objective of determining the optimum production practice.

Keywords: Clean steel, inclusion, deoxidation, ladle metallurgy

ÖZ

ÇEMTAŞ ÇELİK FABRİKASINDA KALINTI KONTROLÜ

Şahin, Berkay
Yüksek Lisans, Metalurji ve Malzeme Mühendisliği Bölümü
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ÇEMTAŞ Çelik Fabrikası, başlıca otomotiv endüstrisi tarafından kullanılan alaşımlı çelikler üretir. Müşterilerin "daha az, daha küçük ve daha homojen dağılmış kalıntı" içeren temiz çelik talepleri her geçen gün artmaktadır. Müşterilerin bu yoğun talepleri ultrasonik test ile kontrol edilmektedir. Bu testlerde tespit edilen hatalı bölgeler kesilerek hurdaya ayrıldığından üretim kaybı oluşmaktadır. Dolayısıyla, asıl konu üretilen temiz çelik ile üretim kayıplarını azaltmaktır. Bu çalışmanın amacı, ÇEMTAŞ tarafından üretilen çeliklerin temizliğini etkileyen faktörleri araştırmaktır. Bu amaçla, çelik üretim pratiği baştan sona gözden geçirilmiş ve nihai üründeki kalıntıların olası nedeni tespit edilmeye çalışılmıştır. Bu bulgulara dayanarak, sıcaklık, zaman, çeliğe eklenen malzeme miktarları, karıştırma şiddeti vb. gibi proses parametreleri optimum üretim pratiğini belirlemek amacı ile değiştirilmiştir.

Anahtar Kelimeler: Temiz çelik, kalıntı, deoksidasyon, pota metalurjisi

To My Dear Family...

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CHAPTER 1

INTRODUCTION

Today in the steelmaking industry, high productivity and cost reduction are becoming necessary with respect to the importance of competitiveness. On the other hand, demands of the steel customers are getting tighter regarding the steel cleanliness and the steel quality from the producers. In order to challenge these demands and competitiveness, defect free steel production comes into prominence for the steel plants. All over the world, great efforts have been performed to optimize steelmaking processes so as to keep defects at the lowest possible level.

ÇEMTAŞ produces several types of alloyed steels in different sections and sizes as raw material mostly used for the automotive industry. The quality of steel produced at ÇEMTAŞ is high but steel products having defects can be come across during production as usually observed in any steel plant. All round bars produced at ÇEMTAŞ are controlled by using non-destructive testing methods, which are magnetic flux and ultrasonic testing, to detect surface, subsurface and internal defects. Laborious grinding operations are required for surface and subsurface defects. Moreover, presence of internal defects necessitates cutting operations, which result in production losses.

This study was carried out with the objective of investigating the origins of defects in ÇEMTAŞ steel products and taking precautions in order to decrease production losses. With this objective, the steelmaking practice used was thoroughly investigated. Samples taken from almost all steelmaking operations and the products were examined and evaluated with microscopic, macroscopic and also non-destructive testing studies.

Results of these studies indicated that one of the causes of defects was non-metallic inclusions. Parameters of electric arc furnace, deoxidation, ladle, vacuum degassing and continuous casting practice were considered as variables to be optimized. Certain measurements were conducted in order to decrease inclusion and hydrogen contents of the steel. After precautions taken for decreasing defects, the measurements showed that these precautions were very effective.

CHAPTER 2

LITERATURE REVIEW

2.1 Steel Cleanliness and Non-Metallic Inclusions

With growing demands of customers, one main aspect in steelmaking industry is the cleanliness of steel. Cleanliness is a crucial concept for steel products due to its influences on mechanical properties of the steel such as fatigue, toughness, formability and ductility. Steel cleanliness is related to characteristics of inclusions that are present in steel. For the past few decades, much effort has been expended on the cleanliness of steel. Degree of cleanliness can vary according to steel grade although the production route of the steel is similar. And also, demands for cleanliness are changed owing to application and usage area of steel product. Nonetheless, size, morphology, distribution and composition of non-metallic inclusions play an important role for each steel product. The formation of inclusions is inevitable during steelmaking but their amount and size can be minimized to as low as possible level which is required for production of clean steel. So, deep knowledge of inclusions is essential for getting an effective steel production which satisfies the specifications of customers and decreases the production losses. At first, this knowledge on inclusions should include the sources and types of inclusions, formation, modification and removal mechanisms, and then the necessary precautions should be taken to optimize the characteristics of inclusions that are mentioned previously.

2.2 Classification of Non-Metallic Inclusions

Non-metallic inclusions can be classified into several groups with regards to many perspectives. Based on the chemical composition of these inclusions, they are mainly divided into three groups, which are oxides, sulfides and nitrides. And, non-metallic inclusions can be exogenous or indigenous depending on their sources [1-3]. There are lots of possible sources of non-metallic inclusions during steelmaking.

Exogenous inclusions can result from steel-slag reactions, atmosphere contact, refractory erosion and some other cases which can be prevented by re-arranging process steps. This means that exogenous inclusions can be eliminated. Ladle slag and refractory can be given as examples of sources of these inclusions.

Indigenous inclusions arise from reactions occurring in the molten or solidifying steel. They begin to form in ladle during deoxidation which will be explained in one of the next sections and they continue to form throughout the entire process including the solidification of the steel. The formation of indigenous inclusions is inevitable but it is possible to decrease their amount to an acceptable level which is essential for production of clean steel. Unlike exogenous ones, these inclusions are usually small in size and hence, exogenous inclusions are more detrimental. The process to remove inclusions is mostly done by stirring of the steel with noble gases or induction, and by degassing of steel in ladle.

2.3 Inclusion Detection and Evaluation Methods

There are several methods to detect inclusions and to evaluate the cleanliness of steel directly or indirectly [4]. It is vital to select a suitable method in order to find out a proper remedy for preventing inclusions and hence, to increase steel cleanliness. Some direct methods are metallographic microscope observation

(MMO) [5], scanning electron microscopy (SEM) [6], optical emission spectrometry with pulse discrimination analysis (OES-PDA) [7-10] and conventional ultrasonic scanning (CUS) [8]. These methods give reliable results about inclusions but indirect methods such as total oxygen method and nitrogen pick-up method are commonly used in steelmaking industry due to cost, time requirements and sampling difficulties of direct methods [4].

Total oxygen method, which is mainly used in the present study, is an indirect measure of steel cleanliness. This method is an indicator of the amount of oxide inclusions present in steel, but the method does not give any information about the size and distribution of oxide inclusions.

2.4 Carbon-Oxygen Correlation in Electric Arc Furnace

In this thesis, it is focused on oxide inclusions quantitatively, most of which form as a consequence of deoxidation and reoxidation processes. Regarding on the cleanliness of steel, oxygen is one of the most important elements in order to be controlled throughout the steelmaking operations. There is a tight correlation between the oxygen content of steel and the alloying elements, especially carbon, prior to deoxidation.

The carbon-oxygen reaction, which is one of the most important and fundamental reactions in steelmaking, takes place in basic oxygen furnaces and electric arc furnaces. The reaction tends to be in equilibrium according to the following reaction:

$$\underline{C}\ (\%) + \underline{O}\ (\%) = CO\ (g) \qquad (1)$$

It is known that in liquid steel the equilibrium constant of the formation CO(g) is:

$$K = \frac{P_{CO}}{a_C \times a_O}, \quad logK = \frac{1168}{T} + 2.07 \tag{2}$$

$$\mathrm{a_C} = \mathrm{f_C} \times \%\mathrm{C}, \quad \mathrm{a_O} = \mathrm{f_O} \times \%\mathrm{O} \tag{3}$$

According to Equations 1, 2 and 3 given above, multiplication of mass percent in steel bath of these elements gives a constant value, which is approximately 0.0020 if the partial pressure of carbon monoxide is assumed to be 1 atm and the temperature is 1600^{o}C [11-13].

In the light of these equations, increasing carbon content in the bath decreases the oxygen level during the meltdown and just before tapping. On the other hand, the oxygen content of the steel tapped into the ladle is usually high with the purposes of slag foaming, refining and energy consumption.

2.5 Steel Deoxidation

It is necessary to lower dissolved oxygen in molten steel, since solubility of oxygen in solid steel is negligibly small and it approaches to zero with decreasing temperature below its melting point. For these reasons, decreasing oxygen solubility of steel may result in oxides, CO bubble formation and also blowholes during continuous casting.

Deoxidation is the first step, where the dissolved oxygen content of steel is reduced, and it is also beginning of inclusion path in entire steelmaking process. The formation of oxides is unavoidable during deoxidation and these oxides are more stable than carbon monoxide. Decreasing the amount of oxygen in the molten steel is achieved by adding elements, which have higher affinity for oxygen than iron. These elements are used as deoxidizer during deoxidation operation.

Oxygen affinity of different elements is given in Figure 2.1. This diagram indicates the oxygen affinity of elements owing to free energy of oxide formation as a function of temperature.

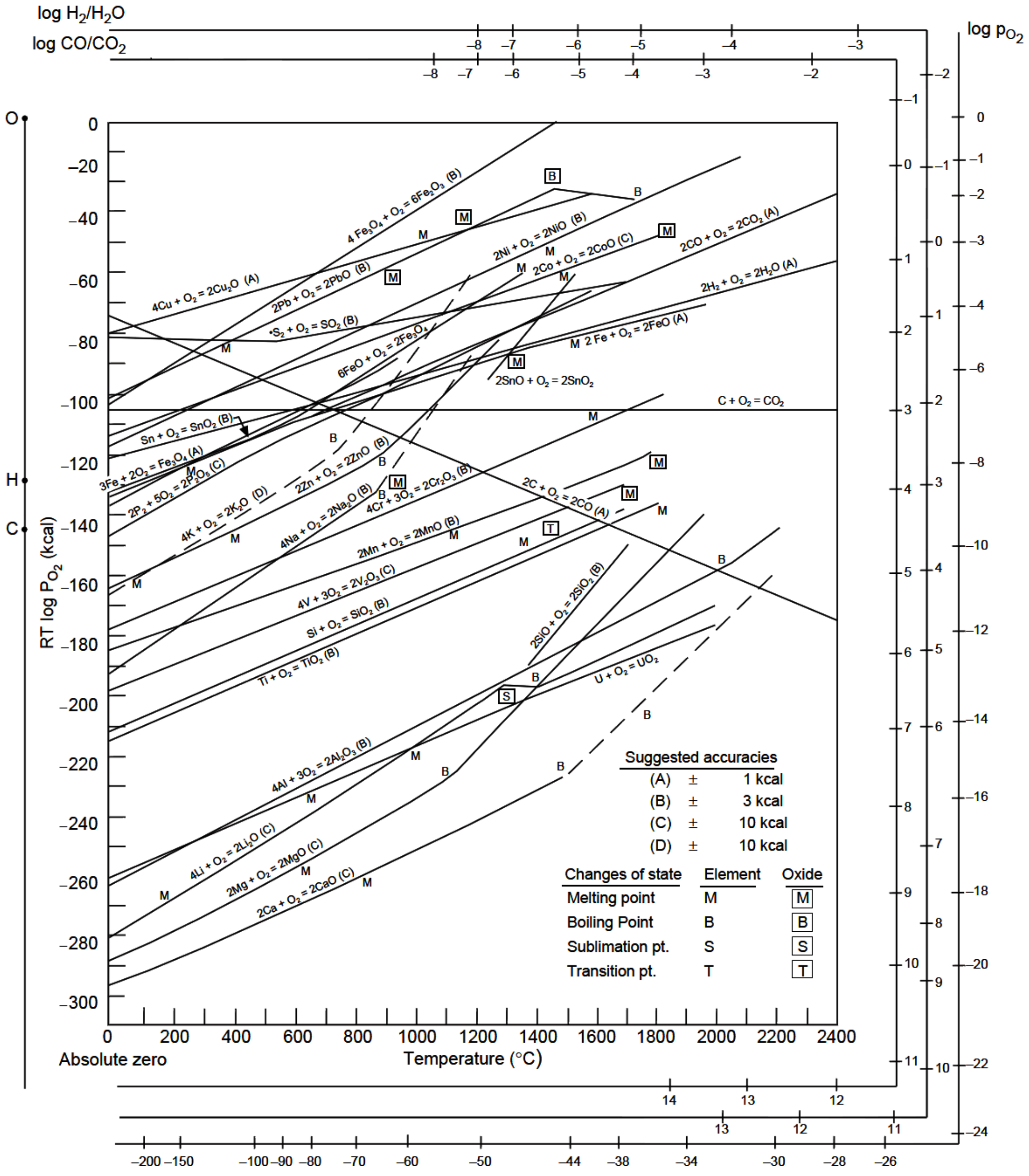


Figure 2.1 – Standard free energy of formation for various oxides [16]

According to the diagram, each element, which has lower free energy of oxide formation lower than that of FeO, may principally be used as deoxidizer. If deoxidizer is added into the molten steel and deoxidation process is performed at a constant temperature, it is called as primary deoxidation and this results in primary inclusions. Deoxidation continues with decrease in temperature and also during solidification. This is called as secondary deoxidation, which results in secondary inclusions [14, 15].

It is also notable that the activity of these elements in solution differs from the case of their pure substances. Figure 2.2 shows equilibrium of some deoxidizers with oxygen and significant differences of their oxide products, namely deoxidizing power of various elements. Among deoxidation elements, zirconium, aluminum and titanium are strong deoxidizers but aluminum, silicon and manganese are mostly used deoxidizers due to cost considerations in practice. Manganese is the weakest, aluminum is the strongest one and silicon is in between when these mostly used deoxidizers are taken into account.

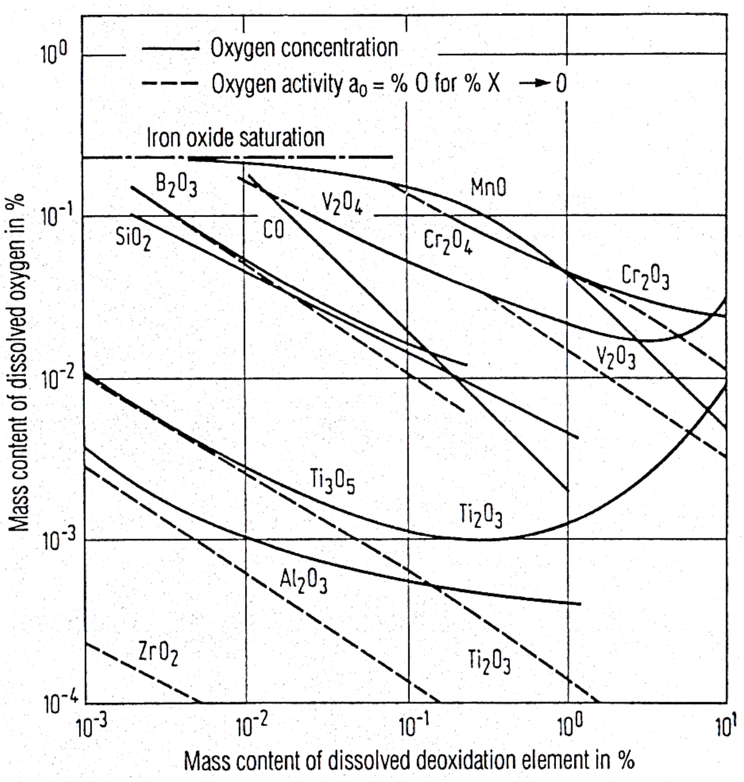


Figure 2.2 – Deoxidation equilibria in liquid iron at 1600^{o}C [17].

2.5.1 Deoxidation with Aluminum

Deoxidation with aluminum is the most common method in the steelmaking industry because of higher affinity of aluminum for oxygen. The following reaction takes place when deoxidation is carried out by aluminum.

$$2\underline{Al}\ (\%) + 3\underline{O}\ (\%) = Al_2O_3\ (s) \qquad (4)$$

This is a well-known reaction between dissolved oxygen and aluminum in the melt. After addition of aluminum into the steel, aluminum melts and it dissolves in molten steel. Dissolved aluminum reacts with dissolved oxygen, and then the reaction yields solid Al_2O_3 deoxidation product, namely primary inclusion. As a result of this deoxidation, low amount of dissolved oxygen in the steel is obtained.

Furthermore, it is possible to get lower soluble oxygen content for Al killed steels by investigating thermodynamics of the Equations 4 and 5, where K is equilibrium constant of Reaction 4.

$$a_O = \left[\frac{a_{Al_2O_3}}{{a_{Al}}^2 \times K}\right]^{\frac{1}{3}} \qquad (5)$$

According to the Equations 4 and 5, dissolved oxygen depends on $a_{\underline{Al}}$, K and a_{Al2O3}. It is proportional to a_{Al2O3} and reciprocal of denominators. Solid alumina products become molten calcium aluminates by addition of Ca during deoxidation; this causes decrease in activity of alumina. Thus, soluble oxygen content of steel is lowered after deoxidation operation as shown in Figure 2.3.

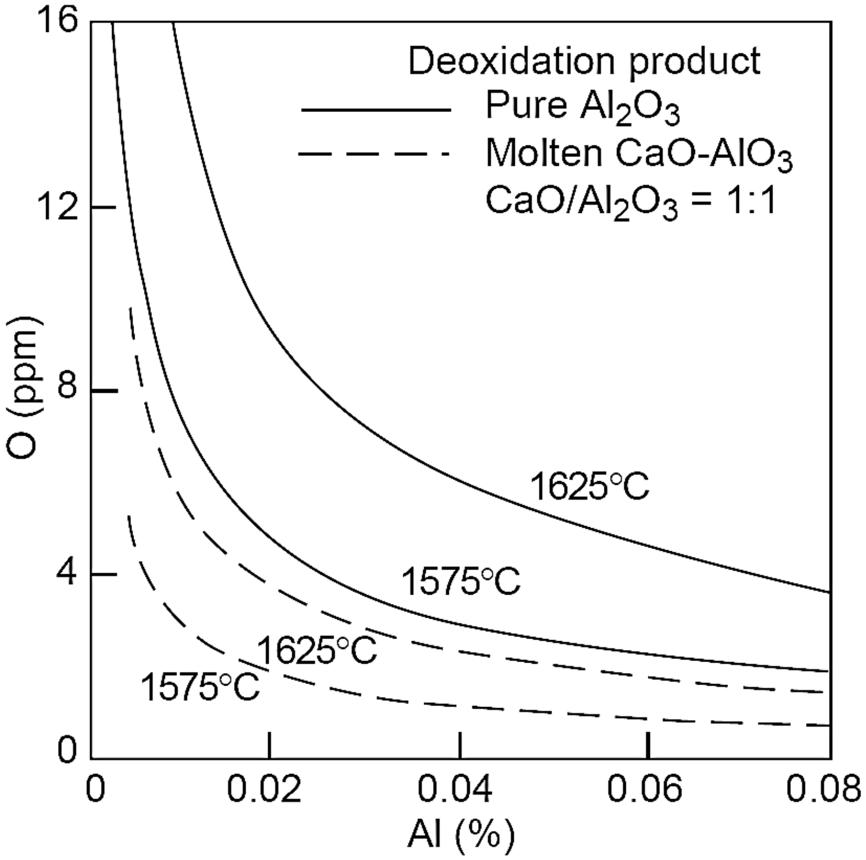


Figure 2.3 – Deoxidation with aluminum in equilibrium with pure alumina or molten calcium aluminate with CaO/Al_2O_3 = 1:1 [13]

2.5.2 The Steps of Deoxidation

It is observed in laboratory experiments and industrial practices that several morphologies of deoxidation products exist in aluminum killed steels and these products can appear in various sizes [6, 18, 19]. Small spherical particles, which can be 1-5 micron in diameter [20], form in molten steel during deoxidation with aluminum. Due to their high interfacial energy, these particles can form clusters by aggregation or collision [21, 22].

In addition to clusters, alumina inclusions may appear as dendrite, platelike and polyhedra [23]. According to numerous studies, it has been proposed that lots of parameters have influence on the growth mechanisms and the morphology of non-metallic inclusions. These parameters are amount of soluble oxygen, the concentration gradients during addition and dissolution of aluminum, the type of stirring and the holding time [24, 25].

In aluminum killed steels, a complete deoxidation includes mainly following steps: dissolution of deoxidizer in molten bath, nucleation and growth of deoxidation products, coalescence and elimination of inclusions from the liquid steel, as it is seen in Figure 2.4.

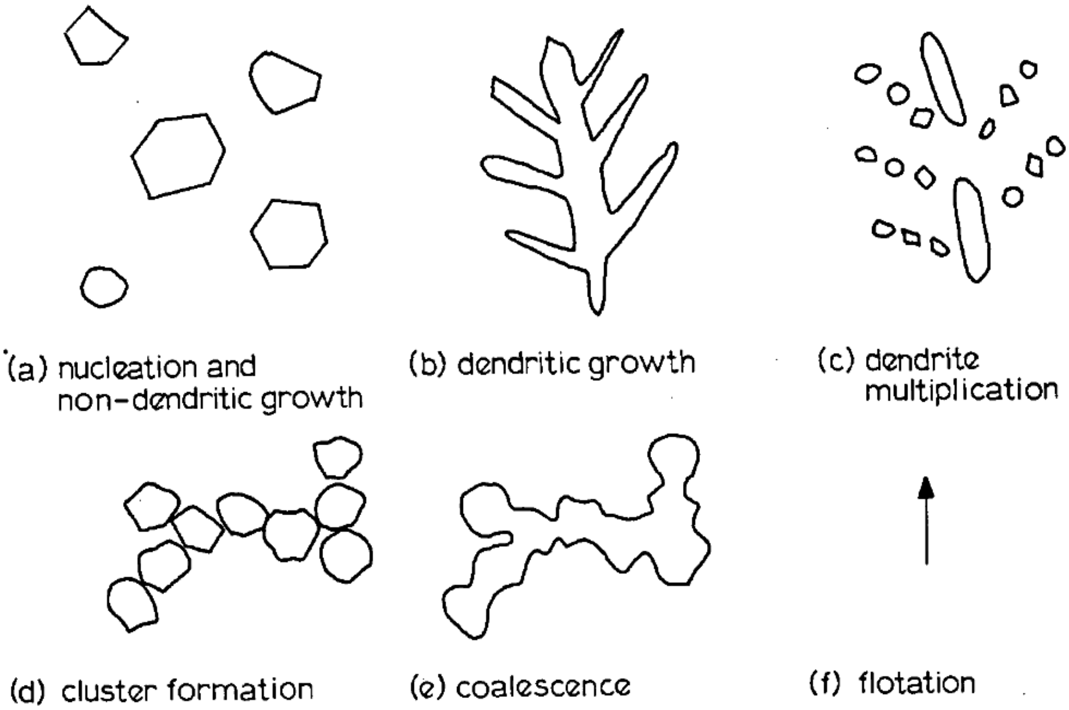


Figure 2.4 – Possible stages of evolution of alumina inclusions [14]

Each of these different stages has its own kinetics and their kinetics is basically related to experimental and industrial circumstances.

Figure 2.5 shows the change in contents of total, dissolved and bonded oxygen with respect to time. According to the figure, the content of dissolved oxygen decreases abruptly and this situation refers to rate of precipitation reaction, which is very rapid [26, 27].

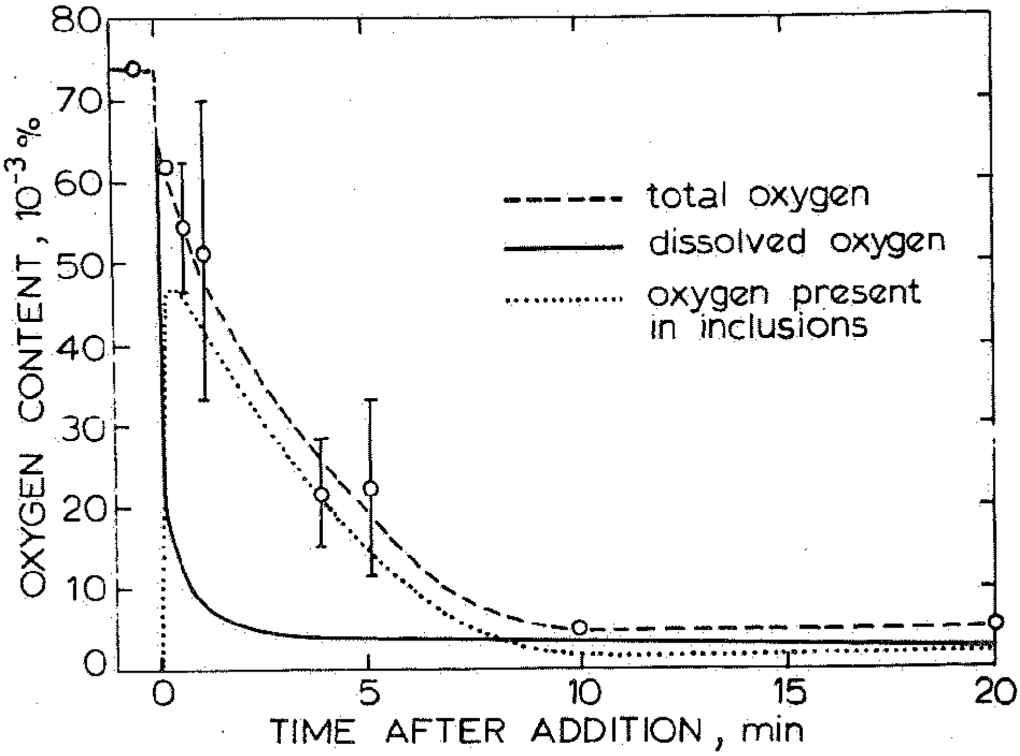


Figure 2.5 – Deoxidation by aluminum at 1600°C [26]

Following the precipitation reaction, coalescence and elimination of inclusions play an important role in order to produce clean steel. According to Gatellier and Olette [26], the majority of oxides are removed from the molten steel within 10 minutes but suspended small particles, which are the rest of floated oxides, need to have long time to rise to the surface. Furthermore, Dekkers et al. [28] have proposed that following the addition of aluminum, the removal of clusters formed during deoxidation process lasts in 15 minutes.

2.6 Elimination of Inclusions

Flotation of inclusions is also one of the major concerns in steelmaking industry. Many laboratory, industrial and modeling studies [29, 30] have been performed about stirring and flotation mechanisms for few decades.

Oxide inclusions have lower density than that of steel, so they usually have a tendency to move towards the surface of the molten steel. According to Stoke's law, rising velocity of spherical particle is proportional to the square of its radius, thus deoxidation products larger in size move faster than smaller ones. Inclusions may collide with each other since inclusions with different sizes rise with different velocities. This mechanism facilitates the flotation. In addition to individual movement of deoxidation products, stirring makes the flotation easier.

Two types of stirring, namely induction and gas stirring are mainly used in industrial conditions. Stirring homogenizes both the temperature and composition of molten steel. And as mentioned, stirring facilitates the elimination of inclusions, as well.

Argon, which is an inert element, is mostly used gas with the purpose of stirring. It can be injected into the bath by using top lance or porous plug installed at the bottom of the ladle.

Besides the method of stirring, its intensity is also important to obtain clean steel. The mixing effect of tapping stream causes vigorously stirring of liquid steel in the ladle and this improves the formation of deoxidation products, then the interaction between inclusions. There are various approaches and equations [31, 32] in order to calculate the stirring power.

Figure 2.6 indicates the relationship between the stirring power and the deoxidation rate constant. According to this figure, deoxidation rate is enhanced with increasing stirring power in the range of industrial practice. After tapping of molten steel into the ladle, there are two types of stirring during refining process due to its intensity. Strong stirring is applied to promote desulphurization and flotation of inclusions. This kind of stirring necessitates attention, since it may cause reoxidation when the stirring is excessively strong. On the other hand, soft stirring is preferentially used in the final stages of steelmaking process to utilize the agglomeration and then floating of inclusions. Hence, appropriate application of stirring intensity and sufficient flotation time facilitates decrease in the amount of remaining inclusions at the end of refining step.

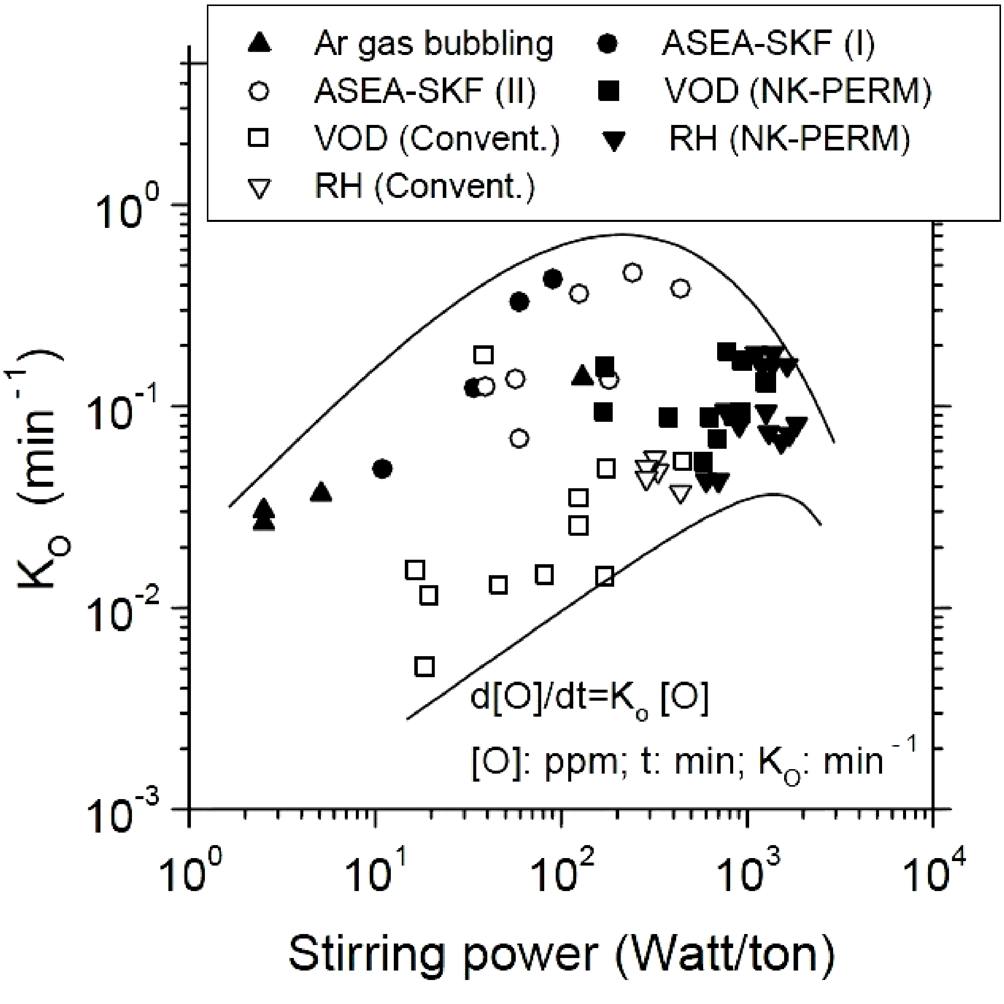


Figure 2.6 – Effect of stirring power on deoxidation rate constant [33]

For the removal of inclusions, characteristics of slag, especially its composition, is a crucial concept to perform absorption and to accelerate the dissolution of deoxidation product in the slag. B. –H. Yoon et al. [34] have reported that the Al_2O_3 absorption ability of slag is able to be optimized by changing its composition. Slag with lower melting temperature have high tendency to absorb Al_2O_3 inclusions. CaO/Al_2O_3 ratio of slag between 1.7 and 1.8 has been found as optimum value to increase ability for Al_2O_3 absorption.

2.7 Calcium Treatment of Steel

Morphology of inclusions, which have not floated, need to be modified and this is generally done by calcium injection. Spherical inclusions are better for mechanical properties. Alumina inclusions are solid at casting temperatures and they have higher contact angle so Al_2O_3 represents non-wetting inclusions in molten steel. Thus, they tend to be easily attached to the nozzle wall during continuous casting of steel. Accumulation of these inclusions on the nozzle wall affects flow rate of liquid steel to the mold undesirably, then they cause nozzle clogging due to adhesion behavior of solid inclusions to the wall.

In aluminum killed steels, calcium is widely used to convert the solid alumina inclusions into liquid calcium aluminate inclusions which form as spherical particles due to surface tension effects. This transformation prevents the nozzle clogging due to the accumulation of solid alumina inclusions. The variation in composition and shape of inclusion is known as the inclusion morphology control.

After the addition of Ca into the molten steel, it melts, vaporizes and then dissolves into the steel. The dissolved Ca or Ca vapor reacts with solid alumina inclusions to yield calcium aluminates. The reaction between Ca and Al_2O_3 forms various calcium aluminates with different melting temperatures as shown in Figure 2.7.

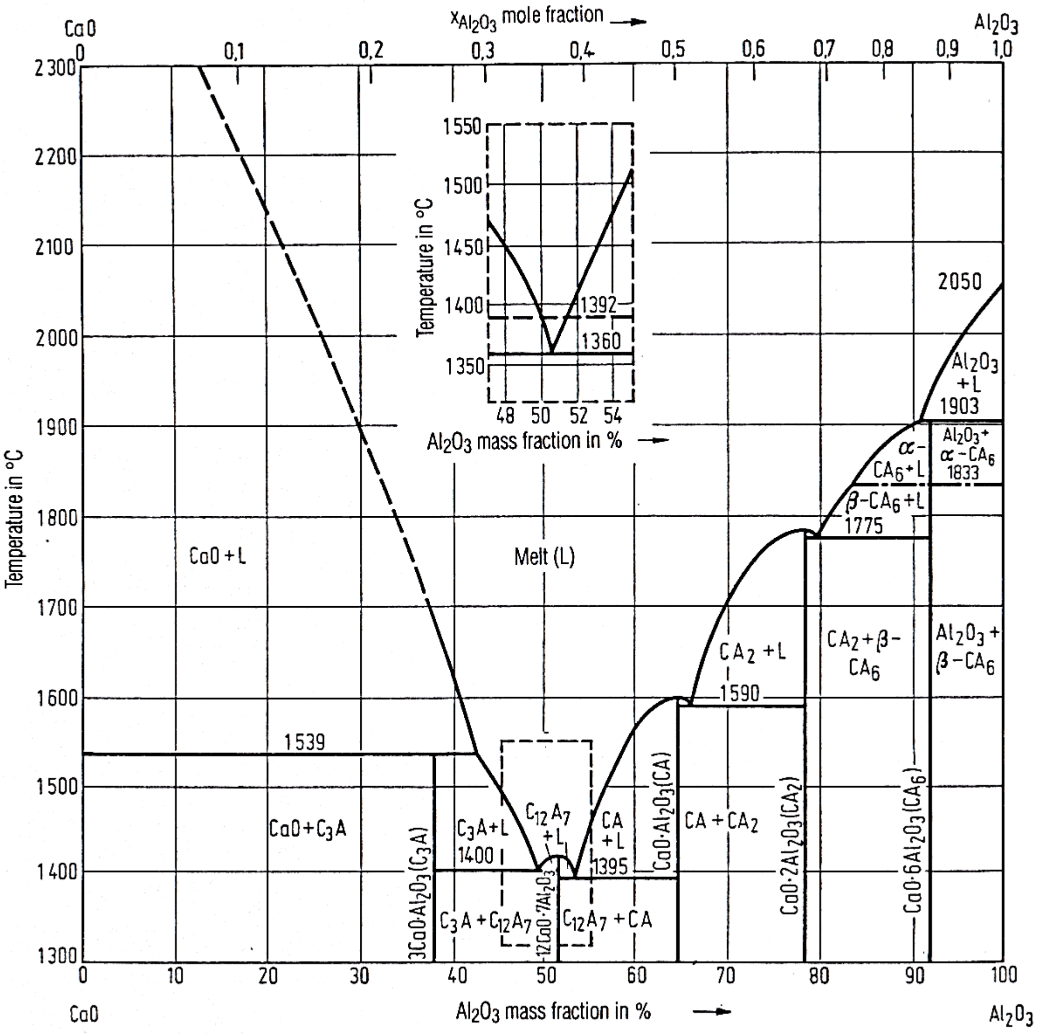


Figure 2.7 – CaO-Al_2O_3 phase diagram [35]

Among these calcium aluminates, $12CaO.7Al_2O_3$ compound has low melting temperature and it is in the liquid state at steelmaking temperature. At this temperature, appropriate additions of calcium into the molten steel is required in order to obtain liquid $12CaO.7Al_2O_3$ compound within narrow limit otherwise solid calcium aluminate products would be formed. It is easier for liquid calcium aluminate inclusions to float out than Al_2O_3 inclusions present as solid in steel, furthermore they do not cause nozzle clogging [36].

For these purposes, calcium is added into the molten steel with two common methods: injection and wire feeding. In the former method, calcium is injected as powder deep into the liquid steel with the help of argon gas through a lance [37]. Alternatively in the latter one, usually CaSi or CaFe cored wire is efficiently fed into the steel by feeding machine depending on the silicon specification of steel grade.

Sulphur is also one of the most important elements for steel cleanliness. It has also an effect on the calcium treatment, which was investigated by Holappa et al [38]. Two different graphs are given in Figure 2.8 below for 0.025%S and 0.010%S. In these figures, calcium and aluminum are used as variables and the intervals of liquid inclusion are shown for 10, 20 and 40 ppm oxygen at 1550^{o}C. It is concluded from the figures that the mentioned intervals shift to lower levels with increasing sulphur content for steels containing certain amount of oxygen and aluminum. In other words, increase in sulphur content leads to required Ca amount, which will liquidize the same amount of aluminum, decreases.

Although perceptible improvements in deoxidation of steel and flotation of inclusions have been obtained, progress must still be made to avoid reoxidation of steel. Presence of large-sized inclusions in the final products is catastrophic and unsolvable obstacle in steelmaking even if they are in limited number. Late reoxidation originated inclusions or those coming from slag or refractory entrainment are not homogenously distributed in the steel.

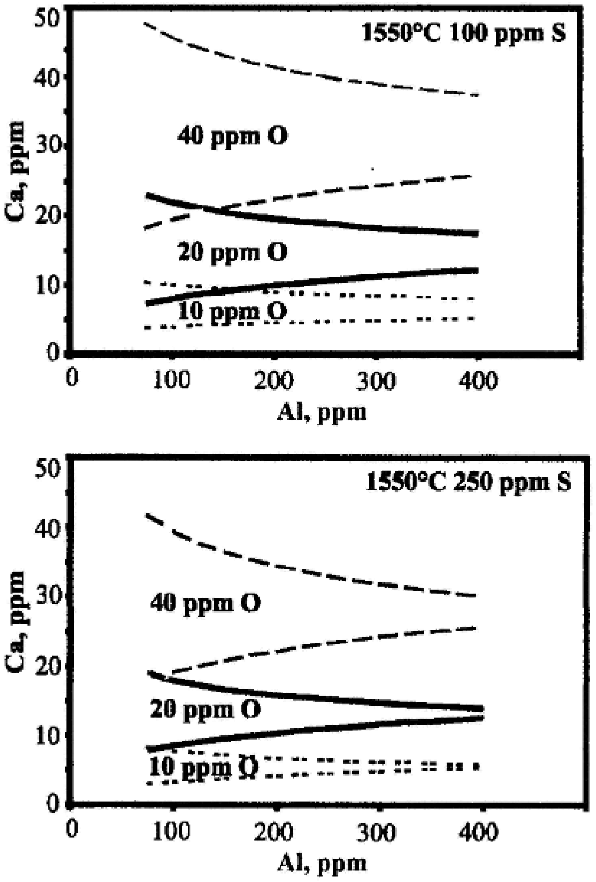


Figure 2.8 – Effect of total oxygen and sulphur on Ca Treatment [38]

2.8 Reoxidation of Steel

Reoxidation is also a major concern in steelmaking due to its detrimental effects on the steel cleanliness and performance since reoxidation products are larger in size compared to the inclusions originated from deoxidation. Reoxidation is the oxidation of deoxidized steel and then reaction of elements present in steel with oxygen, which may come from various sources. These possible sources, which are responsible for reoxidation of molten steel, are mainly air, slag and refractory in steelmaking [39]. Locations of possible oxygen pickup and formation of non-metallic inclusions are illustrated in the Figure 2.9.

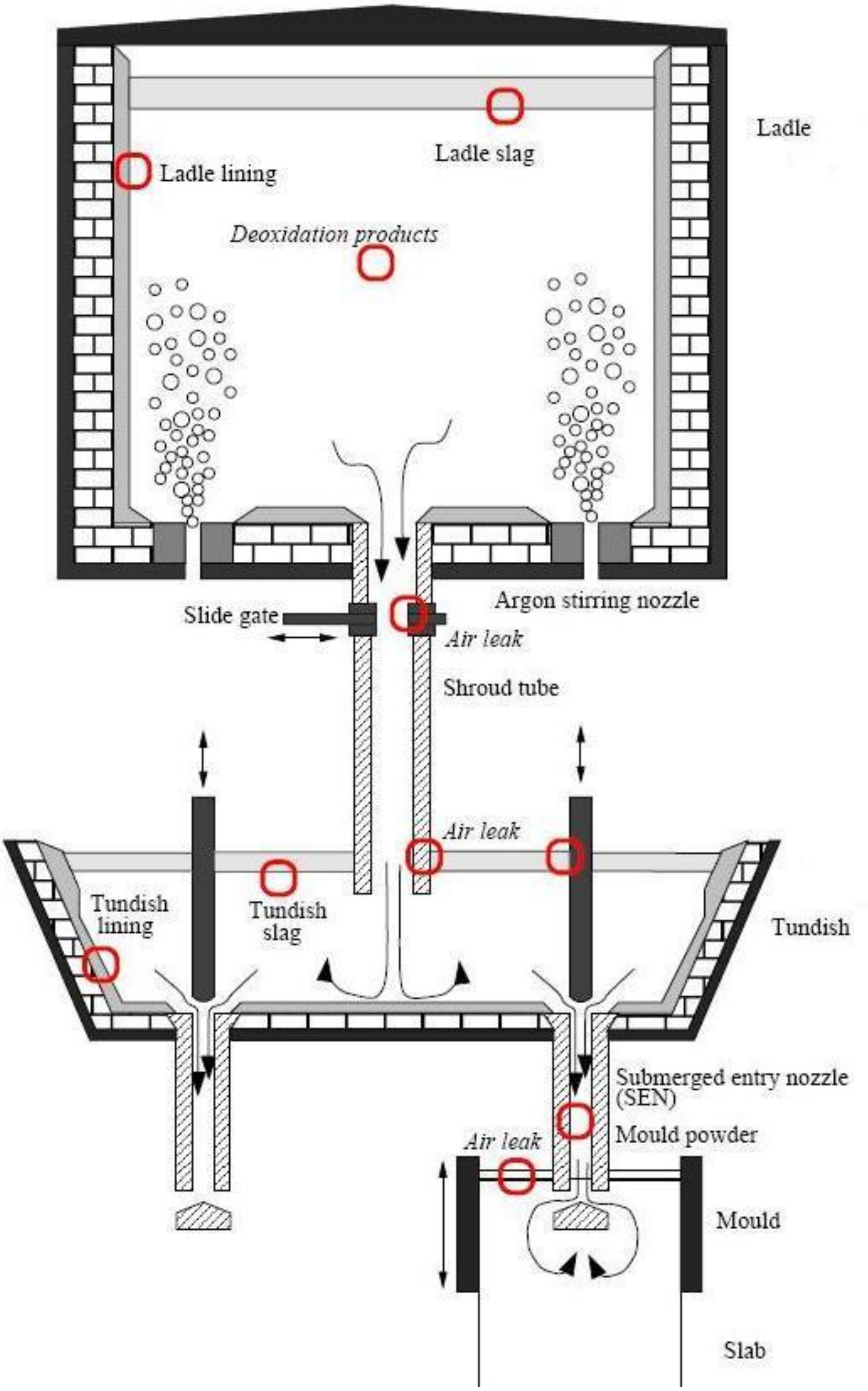


Figure 2.9 – Locations of possible oxygen pickup and formation of non-metallic inclusions [43]

The atmosphere is the most probable source of oxygen since the molten steel comes into contact with air in several stages of steelmaking. The oxygen pickup of steel can vary in the range of 10-1000 ppm [40]. Reoxidation due to atmosphere can be controlled by limiting the exposure of the steel to air.

Interaction between steel and air mainly occurs when tapping of steel into the ladle, pouring of steel into the tundish or molds through air. Air is entrained in the pouring stream as a consequence of these interactions. Besides deoxidizers, carbon is also added into the molten steel during tapping of steel into the ladle. Carbon monoxide formed has valuable shielding effect on the steel and it serves as a shroud for the liquid steel. After completion of ladle refining, a shroud is attached to the ladle and it protects the liquid steel, which flows from the ladle into the tundish. Another shroud attached to the tundish, which is called as submerged entry nozzle or submerged entry shroud according to its mobility, protects steel flowing from the tundish to the mold. Moreover, a gasket made from glass fiber or refractory cloth is fitted at the joints, where steel contacts with air, so as to prevent steel from atmosphere with sealing purposes. Schmidt et al. [41] and Bednarek [42] reported that reoxidation and clogging can be minimized by enhancing argon shielding of the ladle and by improved argon usage in the nozzle wall.

Reoxidation also gives rise to clogging indirectly as follows; firstly new aluminum oxide particles form, and then calcium aluminates are turned into solid aluminum oxide-rich calcium aluminates because of oxygen entrapment into steel.

Reoxidation may take place by metal-slag interactions, as well. Slag is on the top of steel throughout the entire steelmaking process and it may be oxidizing slag with respect to its composition. Oxides such as FeO, MnO and P_2O_5 are considered as reducible compounds and they mostly come from the slag formed in electric arc furnaces. In aluminum killed steels, these oxides in the ladle slag are reduced by aluminum, and that results in reoxidation of steel. Eccentric bottom tapping systems and slag detection systems are designed in order to prevent slag carry-over from electric arc furnace to the ladle.

Refractories are oxide materials and similar to the slag-steel interaction, they keep on contact with molten steel throughout steelmaking processes. Based on this interaction, refractories should be stable oxides, otherwise they will cause increase in oxygen content of steel due to the reducible oxides. MgO-C based refractories are widely used in steelmaking industry. Although MgO is a stable oxide among oxides observed in steelmaking, magnesia may be reduced by carbon under operation conditions. Several studies [44-48] have been conducted on thermodynamics and kinetics of MgO-C reduction. The reduction mechanism may result in additional inclusions, called as spinel (e.g. $MgO.Al_2O_3$). Consequently, these types of bricks may increase oxygen content especially under vacuum operations.

2.9 Vacuum Process

The surrounding atmosphere, charge materials, alloying additions, furnace atmosphere and gas blowing are the common sources of gases to be transferred into the steel. The molten steel comes into contact with all mentioned sources during steelmaking stages. Gas content of liquid steel is vital for many application and usage area of steel since it has a detrimental effect on the final product owing to their limited solubility in the solid state of steel.

According to Sievert’s law, amount of dissolved element such as hydrogen and nitrogen, which is gaseous at room temperature, depends strictly on the square root of equilibrium gas partial pressure.

In order to produce clean steel with regards to gas content, hydrogen and nitrogen contents in molten steel are decreased to as low as possible levels by vacuum degassing.

Similar to vacuum treatment, decreasing the equilibrium gas partial pressure is also achieved with carbon monoxide bubbles in electric arc furnace. The dissolved hydrogen and nitrogen tend to evolve from the steel. Hence, their amount is decreased by the help of CO bubbles. This process is called as flushing. Flushing can also be attained by blowing inert gases.

Hydrogen content during vacuum treatment is controlled not only by the equilibrium gas partial pressure but also by the treatment duration. The effect of vacuum operation on the hydrogen content of steel was studied with respect to time for the vacuum level less than 5 mbar. According to K. N. Jha et. al [49], the vacuum time between 14-17 minutes is required to achieve soluble hydrogen content up to 1.2 ppm with an average of 4.5 ppm beginning of vacuum. The effect of vacuum duration on the dissolved hydrogen content of the steel after the operation is given in Figure 2.10.

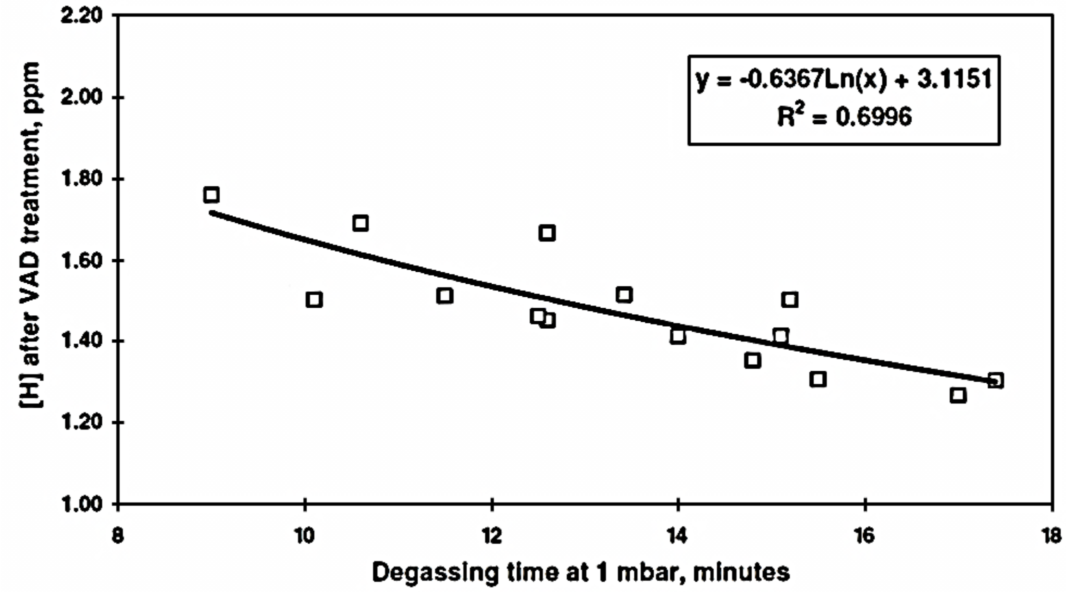


Figure 2.10 – Effect of treatment time under deep vacuum of final hydrogen content of steel [49]

Several additions introduced into the molten steel are likely to react with moisture in atmosphere due to open and wet place in long storage duration. Hydrogen may also come from steel stream and joints, where air contacts with liquid steel. Shrouds and gaskets help to protect steel from possible sources of hydrogen, as well. These additions can vary the amount of hydrogen in molten steel. Hydrogen becomes insoluble when the concentration of hydrogen is too high, thus flaking occurs.

Some elements have also an effect on the solubility of hydrogen in iron. These elements, which increase its solubility in iron, are mostly titanium, vanadium, niobium and chromium, respectively. On the other hand, carbon, boron, silicon and aluminum are the elements that lower the solubility of hydrogen in iron.

Vacuum treatment with regards to oxide inclusions has been studied by many researchers. On the contrary to these researches, Steneholm et al. [50] have investigated change in total oxygen content of steel with time during vacuum treatment.

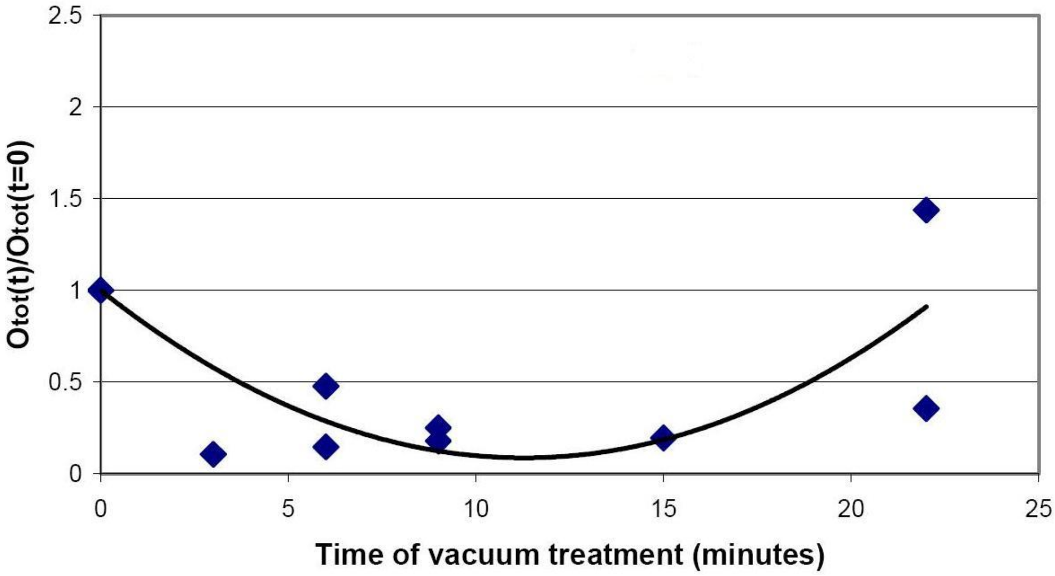


Figure 2.11 – Decrease of total oxygen content during vacuum treatment [50]

As seen in Figure 2.11, it has been found that there is a decrease in oxygen content more than %50 within first 10-15 minutes compared to its total oxygen content before vacuum.

CHAPTER 3

EXPERIMENTAL

3.1 Description of ÇEMTAŞ Steel Plant

Çemtaş Çelik Makine Sanayi ve Ticaret A.Ş. was established at the beginning of 1970’s and it is settled in industrial district on 100.000 m^2 area in Bursa.

Providing the increasing quality steel demand of the market is the primary aim of ÇEMTAŞ. As the technology improved and demand for high quality products increased for the automotive industry, ÇEMTAŞ developed itself by adding new units and improving its older units. These developments consist of establishment a medium section mill with most recent technology, building continuous casting unit and renovating the steel melt shop by changing the EAF with a new 25 ton capacity EAF, establishing a ladle furnace, a vacuum degassing unit and a wire injection unit up to 1990.

In 1995, a new continuous casting machine was founded in order to enhance the cleanliness and quality of steel. Furthermore, there were developments in mill unit. A new medium section mill with descaler and controlled cooling bed was established in 1998.

Today, the capacity of ÇEMTAŞ steel melt shop is 150000 tons of billets/year and the capacity of rolling mill is 210000 tons/year.

ÇEMTAŞ Steel Plant consists of the following units at the present:

- Electric Arc Furnace (EAF) - DEMAG 25 Ton eccentric bottom tapping, water cooled, 18 MVA.
- Carbon injection unit.
- Ladle Furnace (LF) - DEMAG 8 MVA.
- Vacuum Degassing (VD) DEMAG “Capped” type.
- Wire injection unit.
- Oxygen measurement (Celox Lab.II), Hydrogen measurement (Hydris)
- Continuous Casting : CONCAST STANDART AG, S 16-7 CCS

  Two strand casting radius 7-14 m.
  Billet dimensions: 100, 120, 130, 140, 150, 160 mm square.
  Tundish stopper system.
  Mold level control, Billet marking, Electromagnetic stirring.
  Level-2 Quality assurance system (Production quality, Maintenance)

ÇEMTAŞ Rolling Mill consists of the following units at the present:

- Walking hearth furnace – THYSSEN – 50 tons/hour
- Three high mill
- Continuous mill stands
- Orbis bar gauge (diameter measurement)
- Controlled cooling bed
- Cut-to-length unit: abrasive cutting machine and cold shear
- Level-2 quality assurance system

3.2 Steelmaking and Rolling Practice at ÇEMTAŞ

In this section, the production route of ÇEMTAŞ is briefly explained. This explanation includes the units of the plant, equipment used and the process sequence starting with steel melt shop and then continuing with rolling mill.

3.2.1 Electric Arc Furnace

This stage of steelmaking starts with charging of materials to the furnace. It is done in 3 steps with different types of scrap such as domestic, shredded and alloy scrap. The first charge also includes adequate amount of lime. Then, all of the charge is melted completely by creating an arc between the charge and electrodes before introducing oxygen and coke powder. Minimum 0.25% C is oxidized for several reasons which are homogenizing chemical and thermal distribution, decreasing hydrogen and nitrogen amounts by stirring and flushing effect of carbon monoxide bubbles and removing phosphorous from the steel. Moreover, foamy slag is desired so as to use electrical energy more efficiently. To obtain a foamy slag, carbon and oxygen are injected to metal-slag interface. During the entire process after melting in EAF, several samples are taken from the metal and necessary measurements are carried out periodically. After reaching the temperatures 1620-1650 ^{o}C, metal is tapped from furnace to ladle. At ÇEMTAŞ, Electric arc furnace has EBT (Eccentric Bottom Tapping) system, which inhibits the entrance of furnace slag to ladle.

3.2.2 Ladle Furnace

In this unit, mainly refining operations like deoxidation, alloying etc. are conducted. During tapping, deoxidizers such as aluminum and FeSiMn are added into the steel. Then, the deoxidized steel in the ladle is taken to the ladle furnace station. Here, by CaO, CaF_2 and MgO addition a slag with a particular composition is produced. During entire process starting with tapping, the metal is stirred by argon gas, injected from a porous plug located at the bottom of the ladle. Function of this stirring is to make the temperature and composition homogenous and to remove the deoxidation products from the steel. Another case that takes place in ladle furnace is adjustment of composition by addition of alloying elements.

3.2.3 Vacuum Degassing

After completing the process in ladle furnace, the ladle is sent to vacuum degassing unit in order to decrease the amount of dissolved gases. At ÇEMTAŞ, the vacuum is applied under 1 mbar pressure for a predetermined time, which is specified according to steel grades. In vacuum degassing operation in addition to hydrogen and nitrogen contents, the sulphur content of steel is also decreased.

3.2.4 Ladle Furnace II

Ladle is transferred to ladle furnace 2 after vacuum degassing operation is completed for reheating and trimming purposes. By using aluminum, Ca-Si and sulfur wire injection, aluminum and sulfur contents of the steel are adjusted and calcium is added in order to modify inclusions and to improve castability. Finally, heat is sent to continuous casting.

3.2.5 Continuous Casting

The molten steel is firstly taken into the tundish from the ladle by using a shroud that is made up of Al_2O_3 based refractory material. Then, the steel is teemed to the continuous casting molds from the tundish by SES (submerged entry shroud). There is a stopper to control the metal flow rate from tundish to the continuous casting molds.

3.2.6 Rolling Mill

In rolling mill, the route is quite similar for all steel grades. Billets having 140x140 and 160x160 mm sections are first heated to 1250 ^{o}C in furnace for about 2.5 to 3 hours. Then they are rolled to round bars having the diameter of 15 to 80 mm. Temperature of the bars decrease about 1000 ^oC at the end of rolling process and then the bars are cooled slowly in a controlled cooling bed down to 700-800 ^oC, under cover. Finally, the bars are cooled to room temperature in air.

3.2.7 Non-Destructive Testing Unit

At ÇEMTAŞ, all round bars are subjected to non-destructive tests in this unit so as to detect surface, subsurface and internal defects. There are mainly two types of testing machines in non-destructive testing unit. The first machine is circoflux from the company of Foerster. ÇEMTAŞ has three circoflux testing machines and they detect the surface flaws by using method of magnetic flux. The second one is ultrasonic testing machine from the company of Krautkramer. ÇEMTAŞ has also three ultrasonic testing devices with the models USIROB 140 SE, ROB 65 VIS and ROWA B90 PAT. They detect not only surface flaws but also subsurface and internal flaws. Both circoflux and ultrasonic testing machines sprays different colors of dyes to the regions of bars where the flaws are detected. These painted bars are removed separately from the flawless ones and finally, they are examined by macroscopic and microscopic methods in the laboratory.

3.3 Experimental Procedure

Lollypop samples are taken from the molten steel to analyze their chemical composition and carry out measurement of the amount of total oxygen from the same sample. The chemical compositions of these samples were determined by ARL 4460 optical emission spectroscopy with spark excitation. Three pieces of pins, each of which is approximately 1 gram, were prepared from the same sample. The total oxygen contents of these pieces are analyzed by LECO ONH 836, which also determines the nitrogen and hydrogen amount of pins by combustion method. The average of three results is taken into account for total oxygen content of the lollypop sample. Furthermore, the dissolved oxygen in molten steel is determined by Celox sensor containing a Cr/Cr_2O_3 reference from the company of Heraeus Electro-Nite. Both the dissolved oxygen measurement and lollypop sampling are conducted nearly at the same time. At some stages of steelmaking process, the dissolved hydrogen content of the steel melt is determined with the help of Hydris device designed by Heraeus Electro-Nite, as well.

After the rolling operation of billets, round bars are transferred to the non-destructive testing unit in order to detect surface, subsurface and internal defects. As mentioned above, there are two types of machines to determine these defects. Circoflux detects surface defects by generating magnetic current on the bar. The intensity deviations of magnetic currents on the material are detected as surface flaw by the probes in contact with the bar. Moreover, GE IT (Krautkramer) type ultrasonic testing devices detect internal, surface and also subsurface flaws. The orbiting probes within the ROWA device produce ultrasonic sound and this sound wave propagates through the material. The reflected bound beams coming from discontinuities in the bar is determined by the probe and all faulty regions are marked as different color according to the type of defects.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Examination of Defects

ÇEMTAŞ, which is equipped with modern production units, supplies different grades of high quality steels to Turkish industry and international market with the products in different sizes and cross sections. As it is typically encountered in all steel plants, defects can be observed in some of the products. These faulty products are separated from flawless ones by using non-destructive testing machines.

After the tests conducted at NDT unit, samples are taken from the bars that contain internal defects, and then blue fracture test and macro etching tests are conducted on the samples to detect the position and morphology of the flaws.

This study was started by macro etching and blue fracture tests conducted on the faulty region of 30CrNiMo6 steel bar obtained from heat number 4761.

Results of macro etching test conducted according to ASTM E-381 standard and blue fracture test conducted according to SEP 1584 standard are shown in Figures 4.1(a) and 4.1(b), respectively. The detected defects, marked in red circles, are seen to be located in the vicinity of white band, which forms due to electromagnetic stirring. The internal defects observed in ÇEMTAŞ are typically like those shown in Figure 4.1.

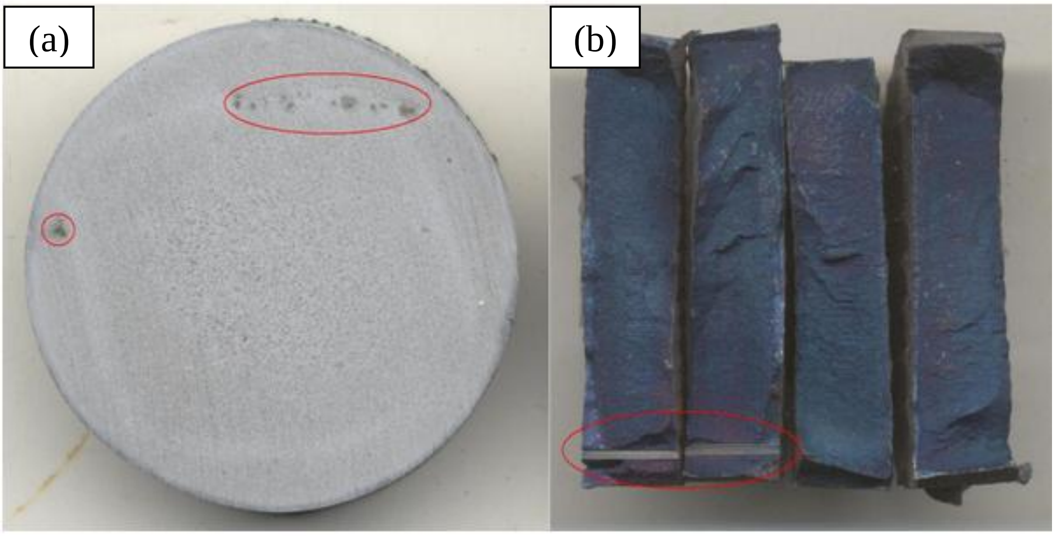


Figure 4.1 – The samples taken from the regions where defects exist (a) Macro etching test result according to ASTM E-381 (b) Blue fracture test conducted according to SEP 1584

Scanning Electron Microscope (SEM) analysis was conducted to the faulty region of the aforementioned sample in Figure 4.1 (b) and the images of defects were taken (Figure 4.2).

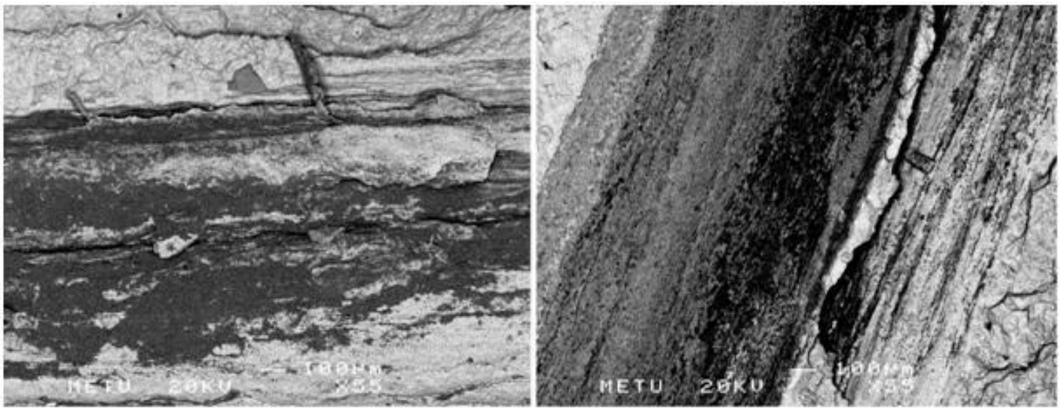


Figure 4.2 – The SEM images of defects marked in red circle at 55x magnification

Two of SEM images that were obtained from different faulty regions of the blue fracture sample are shown in Figure 4.2. Because all faulty regions observed in SEM were similar to those shown in Figure 4.2, no other image is given. Since they are Back Scattered Electron (BSE) images, dark regions show the oxide region and light regions are the steel matrix at 55x magnification. According to the given images in Figure 4.2, oxides in faulty regions are positioned parallel to the direction of rolling and in some regions it is observed that there exists steel matrix in between the oxides that has a structure like one dimensional cluster.

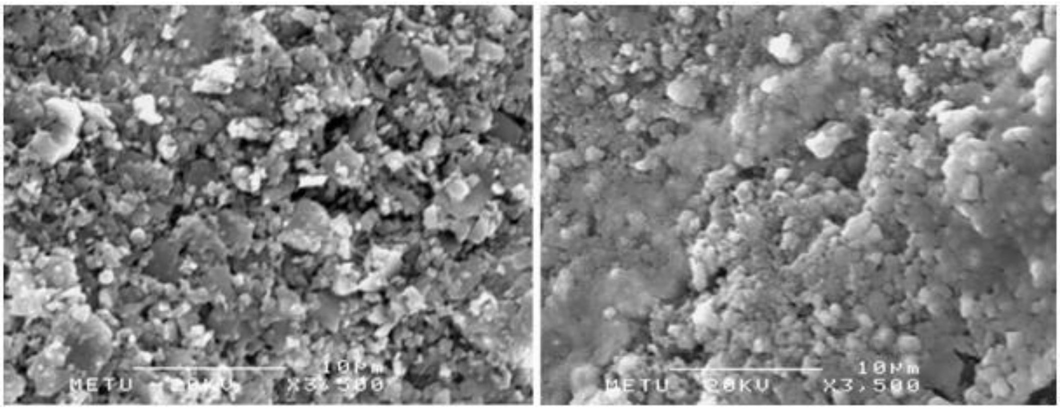


Figure 4.3 – The SEM images of defects marked in red circle at 3500 magnifications

In Figure 4.3, the SEM images of defects that belong to the faulty region are given at 3500x magnification. Here, light clusters are oxides and dark regions are steel matrix. When the images are taken into account, the scale at 3500x magnification is 10 microns and it is observed that the clusters are quite small. Clusters might be formed either by deoxidation or reoxidation of the molten steel. Investigating these images the following results are obtained; if these oxides were formed during deoxidation, they would have floated out until the end of the process, but they could not grow and rise in the steel bath because of insufficient time. According to the images shown in Figures 4.2 and 4.3, it is estimated that they were most probably formed by deoxidation or reoxidation.

Table 4.1 – The SEM/EDX analysis belonging to the spot 1

Element	***Weight Conc %***	***Atom Conc %***	***Compnd Conc %***	***Formula***
O	45.32	59.05	0.00	
Mg	2.02	1.73	3.35	MgO
Al	46.84	36.19	88.50	Al_2O_3
Ca	5.83	3.03	8.15	CaO

Table 4.2 – The SEM/EDX analysis belonging to the spot 2

Element	***Weight Conc %***	***Atom Conc %***	***Compnd Conc %***	***Formula***
O	43.52	58.12	0.00	
Mg	3.37	2.96	5.59	MgO
Al	41.00	32.47	77.47	Al_2O_3
Ca	12.11	6.46	16.95	CaO

Table 4.3 – The SEM/EDX analysis belonging to the spot 3

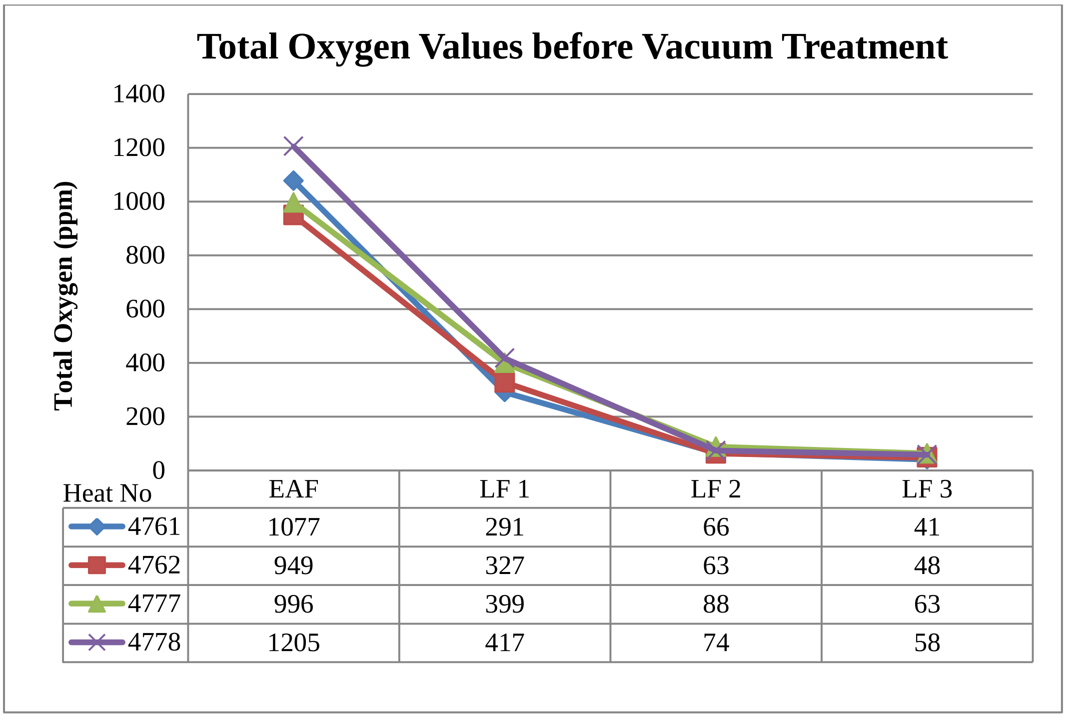
Element	***Weight Conc %***	***Atom Conc %***	***Compnd Conc %***	***Formula***
O	44.70	58.92	0.00	
Mg	0.93	0.80	1.54	MgO
Al	45.66	35.69	86.26	Al_2O_3
Ca	8.72	4.59	12.20	CaO

Three of SEM/EDX analyses conducted to these clusters are given since all analyses were similar to those shown in Tables 4.1, 4.2 and 4.3 in terms of composition. According to analyses, it is observed that they are alumina based oxides which contains approximately 80-90% Al_2O_3 and 10-15% CaO and 3-5% MgO. These oxides do not include Na and Si elements, which are the sign of mold powder since presence of these elements in the inclusions strongly indicates that mold powder has been entrapped by the molten steel during continuous casting. The powder used for mold contains approximately 30% SiO_2 and 10% Na_2O so it can be concluded from these analyses that the source of the inclusions cannot be considered as mold powder. A little amount of CaO was also observed at point analysis of these oxides, which are the result of Ca treatment conducted before continuous casting. Moreover, it might be explained that MgO found in these clusters may come from MgO-C based refractory or ladle slag.

Based on these results, ÇEMTAŞ steelmaking practice was investigated throughout the process considering parameters that have major effects on the formation of oxide inclusions. In order to determine possible sources of these inclusions, at the beginning of this study numerous hydrogen, dissolved and total oxygen measurements were carried out from the various stages of steelmaking. First of all, types and amounts of aluminum deoxidizers were investigated for appropriateness of deoxidation. The correlation between oxygen and carbon was examined in electric arc furnace since it is the first stage to control the oxygen content of steel. The presence of MgO in the inclusions directed this study to the vacuum process and ladle furnace period. Then, in the light of findings, change in total oxygen content during vacuum treatment was investigated with respect to time. Following the vacuum, the effects of the amount of CaSi wire and stirring duration on the extent of defects in the products of the trial heats. Effect of sulphur content and comparison of two ladle shrouds were also studied.

4.2 The Oxygen Measurements at the Beginning of the Study

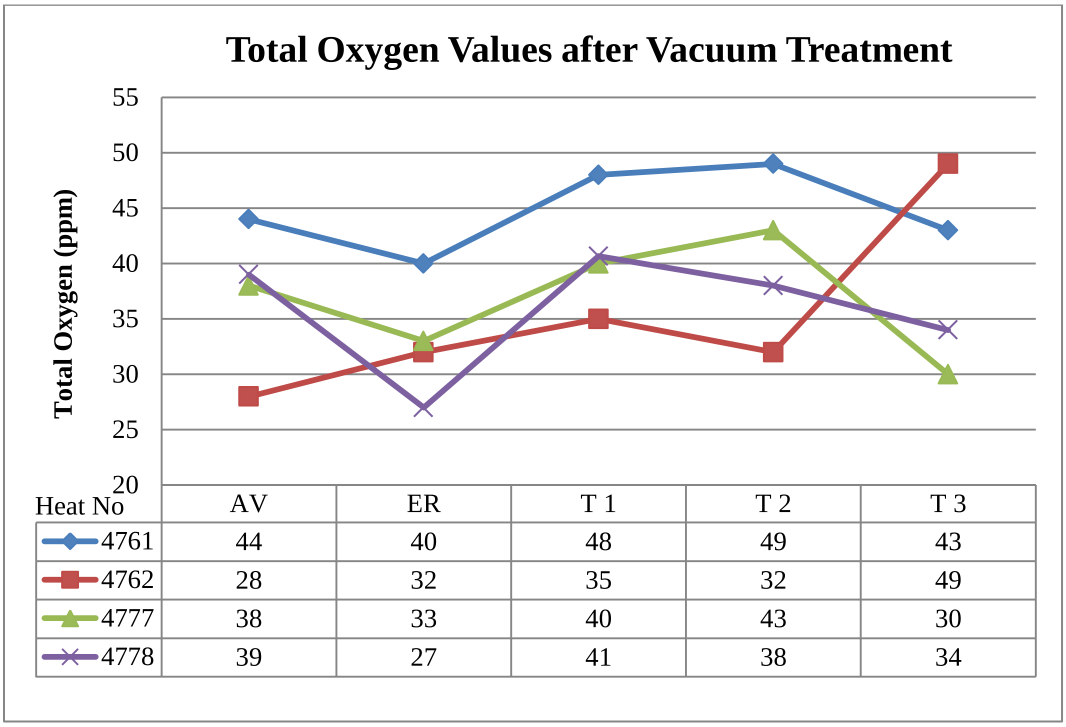
Following the SEM analyses conducted on faulty regions, total and dissolved oxygen measurements were carried out from the various stages of steelmaking during the production of four trial heats. Two of these heats were produced with the chemical composition of 16MnCrS5 according to DIN EN 10084 standard. The other two heats were produced as the steel grade 30NiCrMo8 according to DIN EN 10083 standard. Samples for total oxygen measurements were taken from the molten steel at certain stages like electric arc furnace, ladle furnace, before and after vacuum, the end of refining and tundish. In addition to total oxygen values, nitrogen content of the same samples was measured by LECO ONH 836 device. Dissolved oxygen contents were also measured just after the total oxygen samples were taken from some of the mentioned stages above. The results of total oxygen measurements before vacuum treatment are given as a graph with respect to the heat number and stages in Figure 4.4.



Heat No	EAF	LF 1	LF 2	LF 3
◆ 4761	1077	291	66	41
■ 4762	949	327	63	48
▲ 4777	996	399	88	63
× 4778	1205	417	74	58

Figure 4.4 – Total oxygen values at stages before vacuum treatment for trial heats

According to this figure, total oxygen values continuously decrease for the samples from electric arc furnace (EAF) to ladle furnace (LF) 3; the sample taken from ladle furnace at different times are numbered as LF 1, 2 and 3, respectively. Especially, there is a sharp decrease for the period up to LF 1, which is approximately 2-3 minutes. The rate of decrease of total oxygen decreases at later times as expected. Total oxygen contents of these heats just after vacuum (AV), after refining in the ladle furnace (ER) and in the tundish at different times are shown in Figure 4.5.



Heat No	AV	ER	T 1	T 2	T 3
4761	44	40	48	49	43
4762	28	32	35	32	49
4777	38	33	40	43	30
4778	39	27	41	38	34

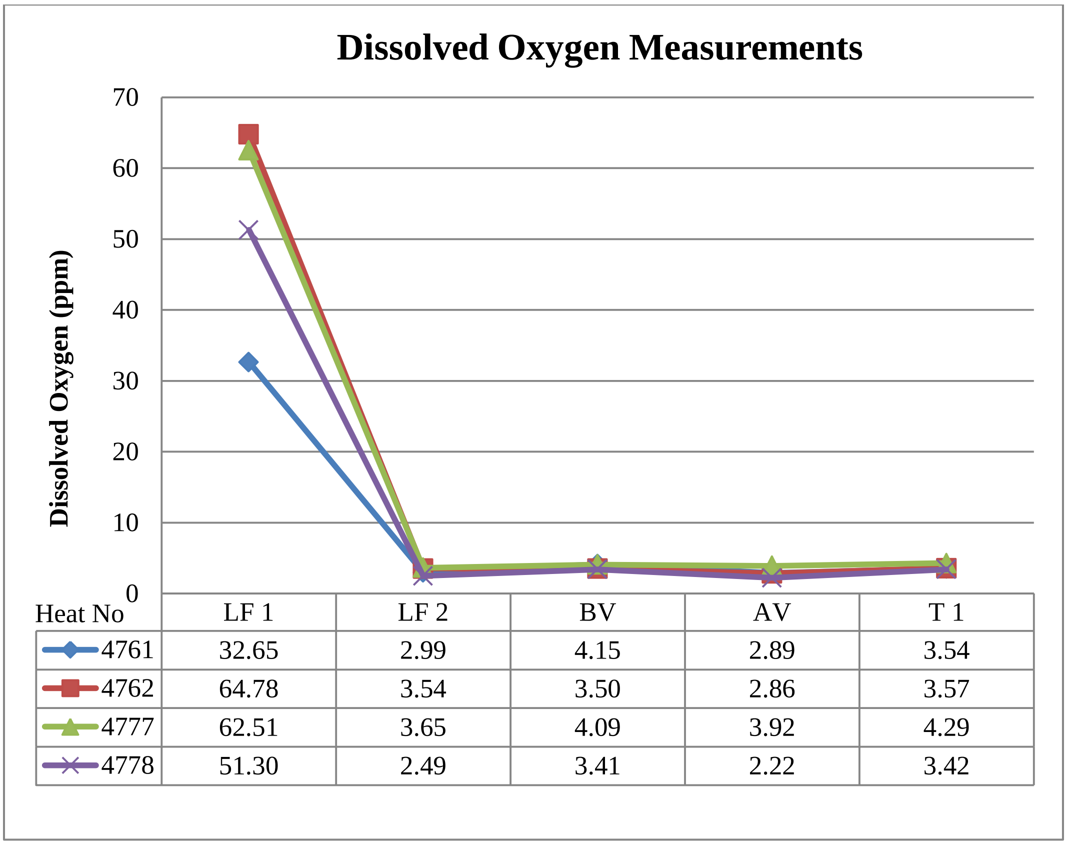
Figure 4.5 – Total oxygen values at stages after vacuum treatment for trial heats

The samples taken from tundish at early, intermediate and later times are numbered as T 1, 2 and 3, respectively. The total oxygen values after vacuum shown in Figure 4.5 are lower than those before vacuum treatment shown in Figure 4.4. A slight increase in total oxygen is observed during the period between the end of refining and tundish. In this period, ladle is opened by slide gate and molten steel flows through the shroud to the tundish. Reoxidation is probable during teeming of molten steel and this can lead to increase in total oxygen values as it is seen in Figure 4.5.

According to SEM images and EDX analyses conducted on samples from faulty regions of the final products, MgO was observed in the inclusions and reoxidation due to MgO based refractory and ladle slag were considered as the sources of these inclusions. But, it has been recently observed that the total oxygen values are higher than the expected values for the stages before vacuum treatment.

For the usual steelmaking practice at ÇEMTAŞ, it is concluded that total oxygen levels before vacuum process are already high for producing clean steel even if there is a possibility of reoxidation during vacuum due to MgO based refractory.

In addition to the total oxygen measurements, dissolved oxygen measurements were conducted by Celox device at certain stages before and after vacuum treatment. The dissolved oxygen contents of the trial heats at the point of arrival to the ladle furnace (LF 1), after addition of a certain quantity of aluminum in the ladle furnace (LF 2), just before vacuum (BV) after vacuum in the ladle (AV) and in the tundish (T1) are shown in Figure 4.6.



Heat No	LF 1	LF 2	BV	AV	T 1
◆ 4761	32.65	2.99	4.15	2.89	3.54
■ 4762	64.78	3.54	3.50	2.86	3.57
▲ 4777	62.51	3.65	4.09	3.92	4.29
× 4778	51.30	2.49	3.41	2.22	3.42

Figure 4.6 – Dissolved oxygen measurements for trial heats

Dissolved oxygen contents of trial heats indicated under the column LF 1 in Figure 4.6 were measured after about 2-3 minutes following the addition of deoxidizers. These soluble oxygen contents are high for an aluminum deoxidized steel which indicates that quantities of aluminum added during tapping were not sufficient which may be due to efficiency of use of aluminum to be low. The soluble oxygen contents (listed under LF 2 and BV) are seen to drop with further additions of aluminum. The soluble aluminum contents are seen to be slightly decreased during vacuum treatment and slightly increased when steel is poured into the tundish. In the light of the results shown in Figure 4.6, it is concluded that deoxidation of the steel was not complete in the ladle due to insufficient amount of aluminum added. High amount of remaining dissolved oxygen absolutely leads to the formation of additional deoxidation products, which may have insufficient time to be eliminated from the molten steel, and these products can cause defects detected by non-destructive testing devices. Thus, it is focused on the usual ÇEMTAŞ deoxidation practice in this study and the practice was completely examined by further oxygen measurements and trial heats.

4.3 The Deoxidation Practice at ÇEMTAŞ

The four trial heats, which were produced to investigate ÇEMTAŞ steelmaking practice, have shown that deoxidation practice used seems to be improper. High amount of dissolved oxygen at the beginning of ladle furnace closely depends on the insufficient amount of aluminum added or the method of aluminum addition into the molten steel. For this reason, the usual deoxidation practice at ÇEMTAŞ was entirely examined by producing additional trial heats.

At first, dissolved and total oxygen measurements were carried out in the production of a heat with the steelmaking practice similar to the previously produced four trial heats. The steel grade of this trial heat was 23MnNiCrMo5-2 according to DIN 17115 standard.

During the production of trial with the heat number 1537, dissolved oxygen measurements were carried out from the molten steel at certain stages like ladle furnace, before and after vacuum, and the tundish. The results of dissolved oxygen measurements are given in Figure 4.7.

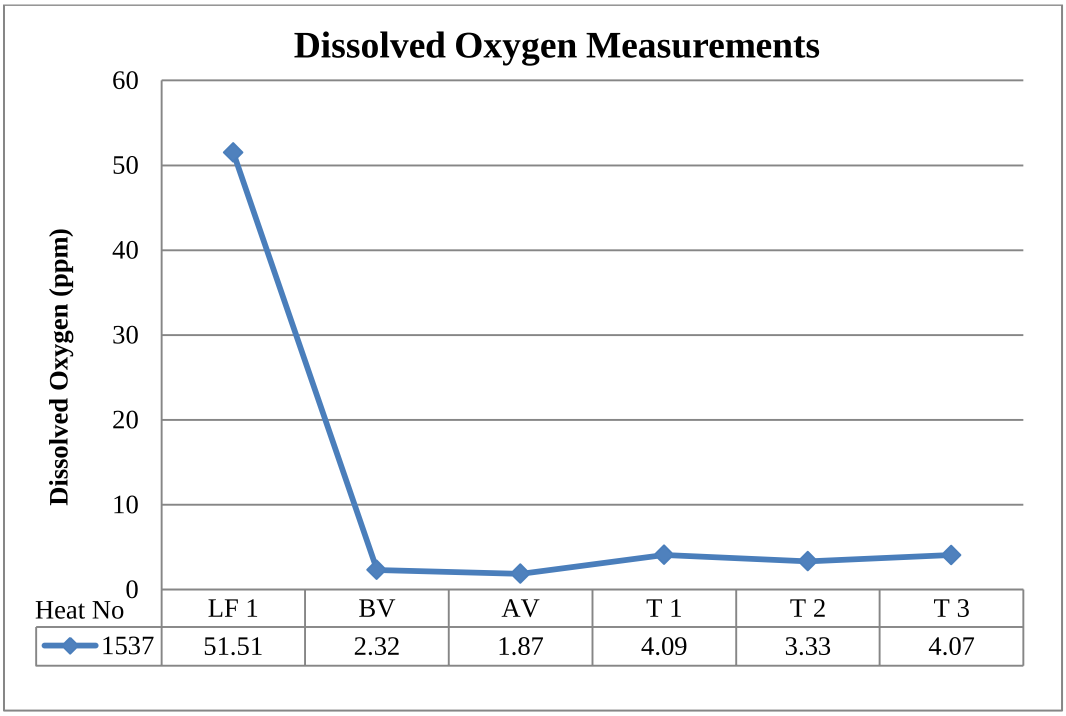


Figure 4.7 – Dissolved oxygen measurements for the heat 1537.

According to this figure, the amount of dissolved oxygen is 51.51 ppm at the stage of ladle furnace 1, which is approximately 2-3 minutes after deoxidation. Similar to the previous trial heats, the amount of dissolved oxygen is also high at this stage for the heat 1537, as well. After this stage, it remains at low levels, which are reasonable for the cleanliness of the steel.

It is considered that the high amount of soluble oxygen at the beginning of the ladle furnace is a result of inadequate deoxidation during tapping of molten steel to the ladle. Then, deoxidation practice used was entirely examined regarding on the amount of aluminum added as deoxidizer.

In this study, further six trial heats were produced in order to investigate the sufficiency of the amount of aluminum, which was increased during tapping to obtain lower soluble oxygen contents just after deoxidation. The amount of FeSiMn formerly added, which also has strong effect on deoxidation of steel, was kept constant during the production of all the trial heats. The carbon contents of the molten steel just before tapping were also nearly the same for these trials. In these six trial heats, amount of aluminum added was gradually increased with respect to the usual ÇEMTAŞ deoxidation practice (UCDP) and dissolved oxygen measurements were conducted just after deoxidation. The results of these measurements where the amounts of aluminum added are expressed in terms of the amount added in ÇEMTAŞ deoxidation practice are tabulated in Table 4.4.

Table 4.4 – Dissolved Oxygen for the Amounts of Al added in terms of UCDP.

Amount of Al added	Dissolved Oxygen
UCDP	69.39 ppm
1.2 x UCDP	28.34 ppm
1.6 x UCDP	5.20 ppm
2.4 x UCDP	4.59 ppm
2.4 x UCDP	1.43 ppm
3.2 x UCDP	2.88 ppm

According to this table, the amount of dissolved oxygen when the quantity of aluminum added is 1.2 times the amount used in the usual ÇEMTAŞ deoxidation practice. There is a noticeable decrease in the dissolved oxygen content of molten steel when the aluminum amount is increased to 1.6 times of the amount used in UCDP. It is also observed that the amounts higher than 1.6xUCDP are sufficient for the lower soluble oxygen values. Hence, these improvements on the deoxidizer amount during tapping satisfies the completion of deoxidation. Furthermore, considering the amounts of aluminum added during deoxidation, the results of dissolved oxygen measurements for six heats were compared with the results obtained from previously produced five heats. These results belonging to eleven trial heats are illustrated in Figure 4.8.

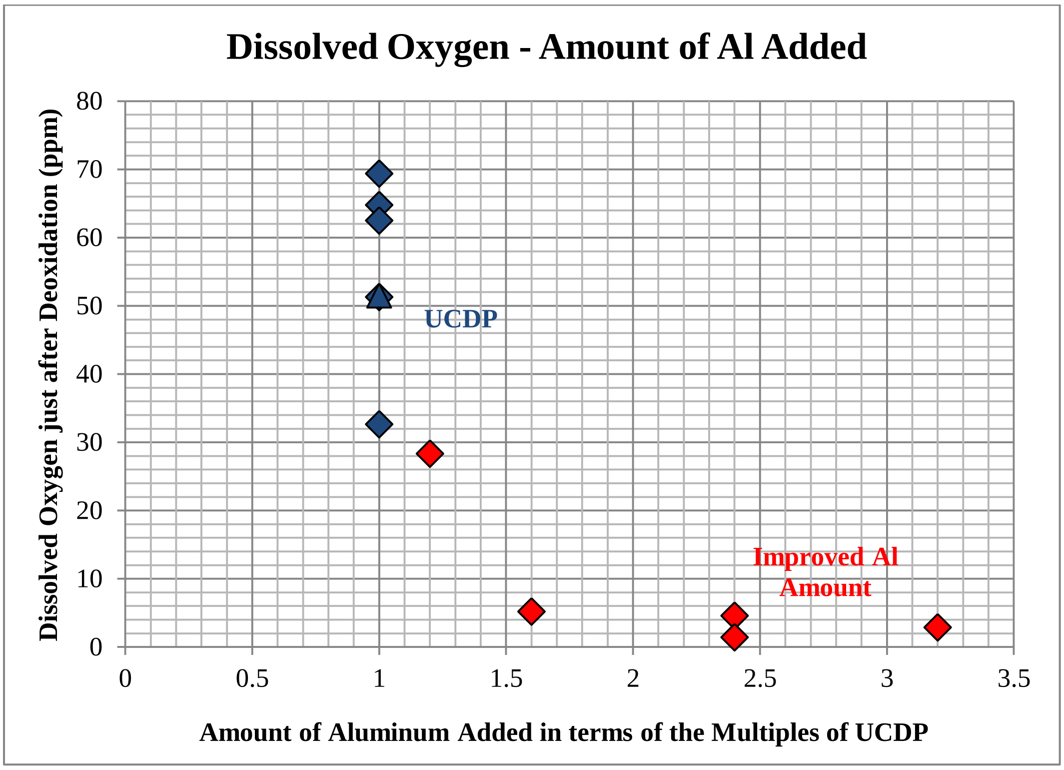


Figure 4.8 – Dissolved Oxygen for the Amounts of Al added in terms of UCDP.

According to this figure, dark blue and red data are shown for the usual ÇEMTAŞ deoxidation practice and increased aluminum amount, respectively. It is obvious that amount of aluminum added in UCDP is insufficient to get low soluble oxygen values just after deoxidation and this amount should be increased to at least 1.6 of the usual practice. In this figure, it is also observed that there are some deviations of dissolved oxygen values for the same amount of aluminum added. It is also known that efficiency of aluminum as deoxidizer can change approximately in the range of 15% to 80% in industrial production. This changing efficiency of aluminum is decided to be the cause of the deviations observed in the figure for the same aluminum addition. To decrease these deviations, aluminum lumps should be added to steel in such a way as to have the aluminum lumps directly under the steel stream during tapping.

Consequently, the main purpose must be that deoxidation should be completed during tapping and any additional aluminum should not be added into the molten steel. By doing so, almost all the dissolved oxygen will be reduced by aluminum during tapping and vigorous stirring during tapping facilitates the elimination of inclusions due to the collisional growth. Except for reoxidation, formation of additional inclusions, which have shorter time to be eliminated from the molten steel, is prevented due to the lower dissolved oxygen during ladle furnace.

On the other hand, total oxygen measurements were conducted at certain stages for the trial with the heat number 1537. The results of these measurements are shown in Figure 4.9.

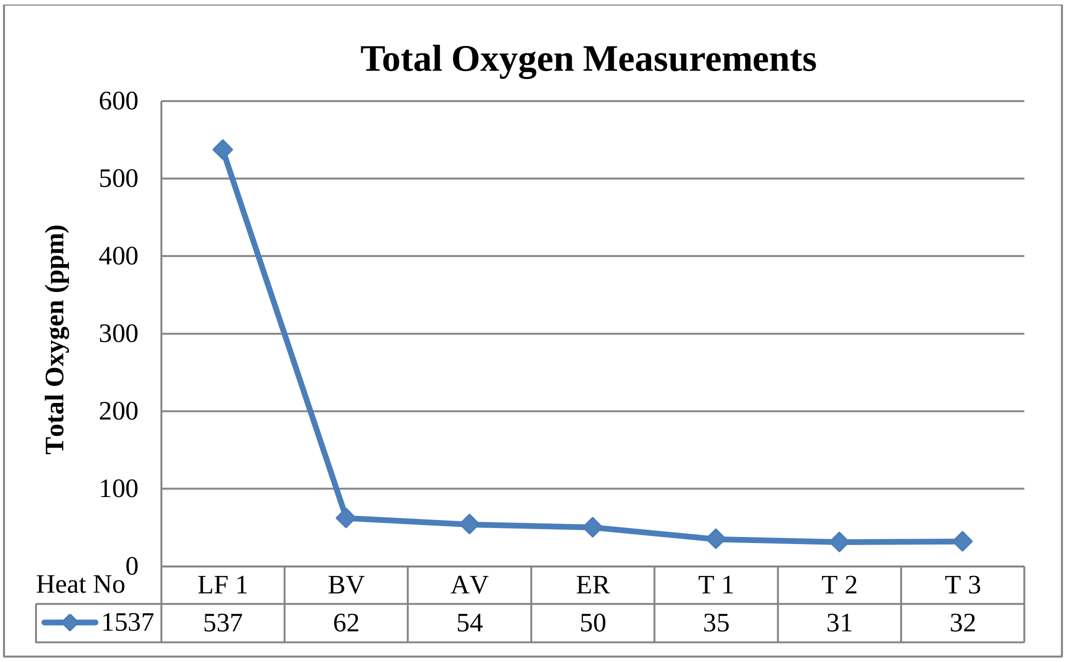


Figure 4.9 – Total oxygen measurements for the heat 1537.

According to this graph, there is a rapid drop of total oxygen from the ladle furnace to the stage before vacuum, and then it decreases slowly until the tundish. This behavior of total oxygen at the beginning of ladle furnace is similar to the case observed in Figure 4.4. In addition to high amount of dissolved oxygen in molten steel, excessive total oxygen content of the steel is also observed at the stages of ladle furnace and electric arc furnace.

Tapping of molten steel with the high oxygen content results in exorbitant amount of deoxidation products and elimination of these products becomes difficult in industrial steelmaking conditions.

Prior to the vacuum process and ladle furnace period, oxygen content of molten steel in electric arc furnace was examined by further oxygen measurements at this stage. Moreover, it is known that the amount of dissolved oxygen before deoxidation strongly depends on the carbon content of molten steel so the relation between these two elements was investigated.

4.4 The Oxygen Content of Steel in Electric Arc Furnace

It is vital for the oxygen content of steel to be low when the inclusion amount in the steel is taken into account. The oxygen content is mainly lowered during deoxidation process, however it is also required to be lower before deoxidation in order to obtain lower amount of deoxidation product.

As it is already known that each furnace has its own characteristics with respect to the thermodynamics and kinetics considerations so it is important to determine the relationship between the oxygen and carbon contents of steel at the tapping stage from EAF to ladle furnace. In order to make correlation between carbon and oxygen contents of steel, total oxygen amount was measured since the device was not available to determine soluble oxygen content of steel in EAF unit. By doing so, it is assumed that all of soluble oxygen in the steel is transformed into oxide form due to decreasing solubility of oxygen with temperature during sampling. With this purpose, the samples belonging to the heats of round rolling campaign were taken just before tapping and the total oxygen values were measured by LECO ONH 836 device.

As previously mentioned in Equation 1, due to the C-O equilibrium in electric arc furnace, carbon and oxygen react and they form carbon monoxide gas according to the following equation:

$$\underline{C}\ (\%) + \underline{O}\ (\%) = CO\ (g) \tag{1}$$

It is assumed that tapping temperature is 1680^{o}C, activity coefficients of carbon and oxygen are 1, and the pressure of CO is 1atm. The following relationship between C and O is obtained by calculating Equations 2 and 3:

$$\underline{C}\ (\%) \text{ x } \underline{O}\ (\text{ppm}) = 21.48 \tag{6}$$

Considering the carbon contents of steel in the light of this theoretical information, samples were taken from twenty heats in electric arc furnace stage and total oxygen measurements were carried out and evaluated. Total oxygen measurements at each carbon level in the range of 0.03%C and 0.25%C were conducted and the relationship between the oxygen and %C was investigated. In addition to this range, measurements were carried out for heats with 0.30%, 0.38% and 1.25% tapping C content in order to observe the oxygen contents of the heats with higher tapping carbon content.

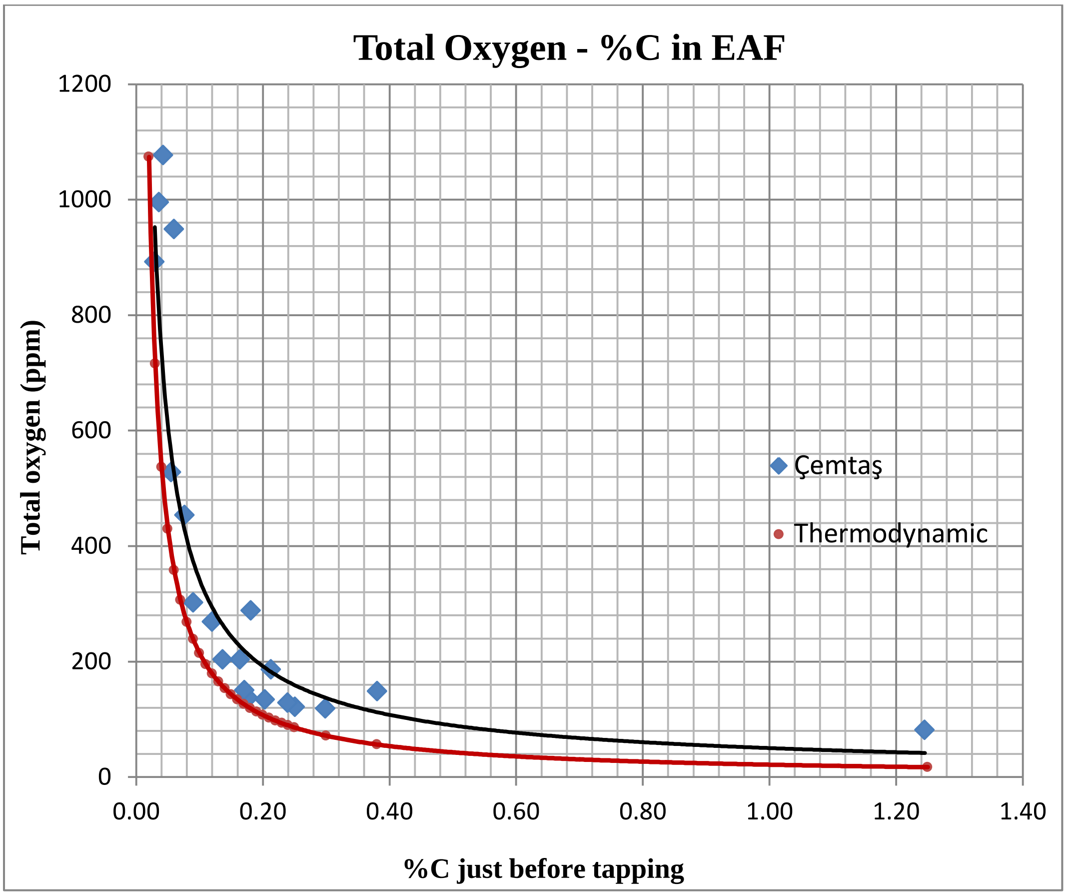


Figure 4.10 – %C and total oxygen values theoretically calculated and measured at ÇEMTAŞ

Based on the results of these measurements, Figure 4.10 is plotted and it is accomplished that these results are similar to the results of theoretical calculations. In order to make comparison, the theoretical C-O equilibrium data and the C-O equilibrium data of ÇEMTAŞ Steel Plant practice are colored as red and blue, respectively.

When this graph was investigated, it was seen that the oxygen contents that correspond to actual carbon content are higher than the thermodynamic equilibrium oxygen contents. However, Pco value is higher than 1 atm in practice, taking this value as 1 atm explains why the oxygen measurement results are higher than the theoretical ones.

While formulating the relationships between carbon and oxygen contents, the fact that tapping carbon is usually not higher than 0.30% is taken into account and the values that are deviating much from the theoretical values are eliminated.

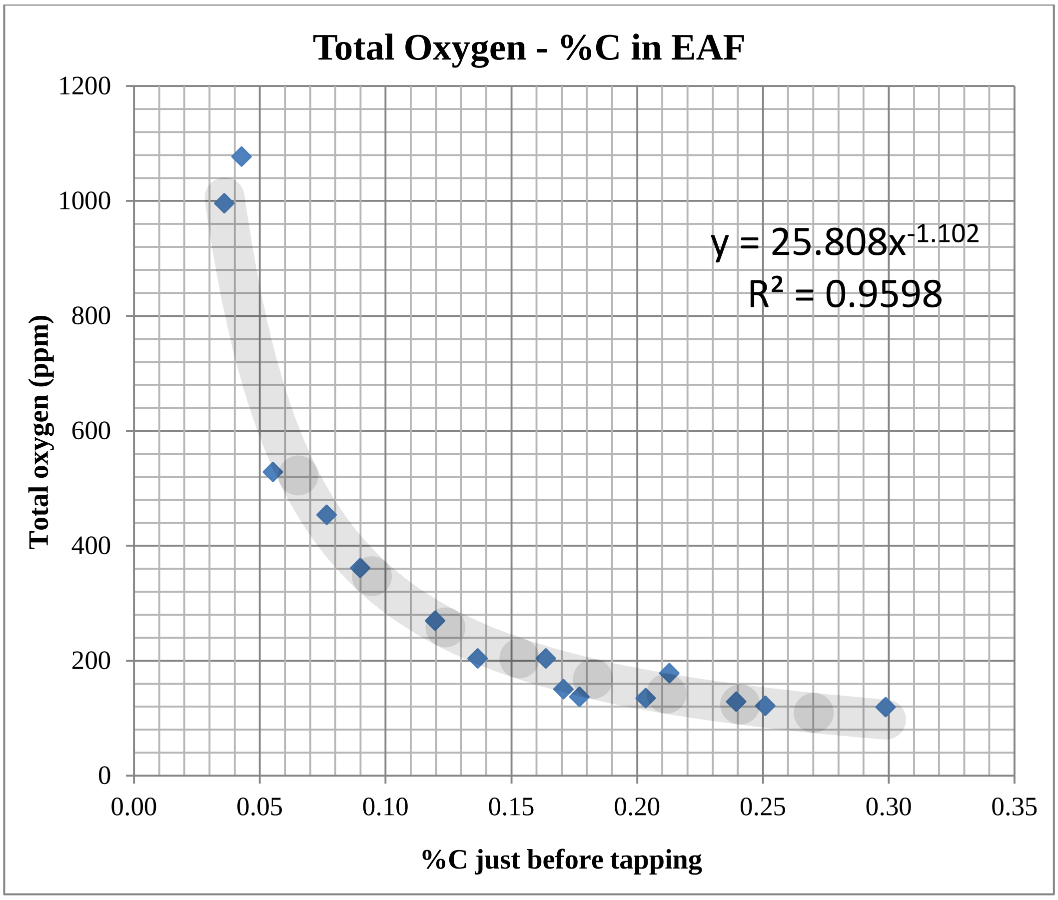


Figure 4.11 – Total oxygen values for %C lower than 0.30 at ÇEMTAŞ

Disregarding the values with high deviation and the values that are higher than 0.30 %C content, Figure 4.11 is drawn using results from test applied to 15 samples. The formula of best fitting curve gives the information about the relationship between C and O content in EAF of the ÇEMTAŞ steelmaking practice:

$$O\ (ppm) \times [C\ (\%)]^{1.102} = 25.808 \qquad (7)$$

As it can be seen from Table 4.5, the oxygen content calculated from the formula is close to the value measured from samples.

Table 4.5 – Calculated and measured total oxygen values at ÇEMTAŞ

% C	Total Oxygen (ppm)	
	Measured	Calculated
0.0360	996	1006
0.0429	1077	829
0.0554	528	626
0.0767	453	437
0.0901	361	366
0.1199	269	267
0.1367	204	231
0.1638	204	189
0.1708	150	181
0.1772	137	174
0.2035	134	149
0.2130	178	142
0.2395	128	125
0.2511	121	118
0.2989	119	98

Thus, the oxygen content of steel just before tapping can be estimated from Equation 7 without any oxygen measurement using the analyzed carbon content. It is obvious that tapping steel with high carbon content is important to obtain low amount of deoxidation product, which has to be eliminated later. Additionally, carbon boiling, which is the result of CO bubbles, is required in electric arc furnace. Bubbling of CO gas flushes dissolved hydrogen and nitrogen from the molten steel. And, it also provides temperature and composition uniformity of the steel bath due to agitation function of these bubbles. It should be performed for the cleanliness of steel.

4.5 The Oxygen Content of Steel during Vacuum Degassing

MgO was observed in EDX analyses of the inclusions in the faulty regions of the products, as mentioned before. Presence of MgO in the inclusions may be due to reoxidation associated with reduction of MgO in the refractories during the vacuum process. To gain insight to possible reoxidation during vacuum treatment total oxygen and aluminum oxide contents of the 4 trial heats were determined before and after vacuum treatment; Total oxygen contents of the samples were determined by LECO ONH 836 and the amounts of aluminum oxide in the same samples were determined by ARL 4460 optical emission spectroscopy. The results of these measurements according to trial heats are given in Table 4.6.

Table 4.6 – Changes of total oxygen and aluminum oxide before and after vacuum

	Total Oxygen (ppm)		**Aluminum Oxide (ppm)**	
Heat Number	Before Vacuum	After Vacuum	Before Vacuum	After Vacuum
4761	41	44	65	22
4762	48	28	76	21
4777	63	38	76	17
4778	58	39	70	31

Aluminum oxide contents of the trial heats are seen to decrease significantly during vacuum treatment from Table 4.6 but decreases in the total oxygen contents of the heats during vacuum treatment are not as significant; in fact for heat number 4761 there is almost no change in the total oxygen content. These results imply that significant amounts of aluminum oxide inclusions are removed from liquid steel but non-Al_2O_3 inclusions form due to reoxidation during vacuum treatment.

The extent of reoxidation during vacuum treatment is expected to depend on vacuum duration. With this in mind, vacuum duration was decided to be decreased. Vacuum duration is very important for hydrogen removal. Effect of vacuum duration on hydrogen removal was therefore investigated by measurement of hydrogen contents of heats subjected to vacuum for different durations. The results of these measurements are tabulated in Table 4.7 where the heat numbers together with steel grades, durations of vacuum, vacuum pressures and the measured hydrogen contents are given. Some heats were subjected to vacuum for a certain period, their hydrogen contents were measured and they were subjected to vacuum again; these are indicated in Table 4.7. Hydrogen contents of several heats were measured before vacuum treatment in previous studies and were found to be in 7.5-8.5 ppm; hydrogen contents of the heats taking place in Table 4.7 before vacuum treatment were therefore not measured.

Table 4.7 – Vacuum trials regarding on hydrogen content, duration and pressure

Heat No	Steel Grade	Hydris (ppm)	Duration (min)	Pressure (mbar)	Vacuum Statement
3361	51CrV4	1.3	14	0.67	Once vacuumed
3362	51CrV4	1.1	12	0.41	The first vacuum
3362	51CrV4	1.3	17	0.45	The second vacuum
3363	55Cr3	1.9	5	0.50	Once vacuumed
3394	51CrV4	1.1	17	0.39	Once vacuumed
1537	23MnNiMoCr5-2	1.2	20	0.20	Once vacuumed
1777	30CrNiMo8	1.7	24	0.24	Once vacuumed
3515	55Cr3	1.8	5	0.33	Once vacuumed
3516	55Cr3	2.3	5	0.27	Once vacuumed
3530	50CrV4	1.3	18	0.25	Once vacuumed
3531	50CrV4	1.0	18	0.18	Once vacuumed
3532	51CrV4	1.3	10	0.26	The first vacuum
3532	51CrV4	1.2	15	0.46	The second vacuum
3616	55Cr3	1.2	9	0.22	The first vacuum
3616	55Cr3	1.0	15	0.26	The second vacuum
3717	30CrNiMo8	1.0	22	0.22	Once vacuumed
3731	42CrMo4	0.9	18	0.17	The first vacuum
3731	42CrMo4	1.2	20	0.49	The second vacuum
3732	42CrMo4	0.8	16	0.19	The first vacuum
3732	42CrMo4	1.0	20	0.21	The second vacuum
3807	34CrNiMo4	0.8	20	0.23	Once vacuumed
3905	42CrMo4	0.7	20	0.29	Once vacuumed

The results listed in Table 4.7 are presented graphically in Figure 4.12 where the hydrogen contents of heats subjected to vacuum for times in the range of 5 to 24 minutes are plotted versus time. The results clearly indicate that hydrogen content of steel subjected to vacuum decreases with increase in vacuum duration. The results also indicate that hydrogen content of steel can be decreased to about 1.2 ppm in about 10 minutes and that decrease in hydrogen content is slight for longer times.

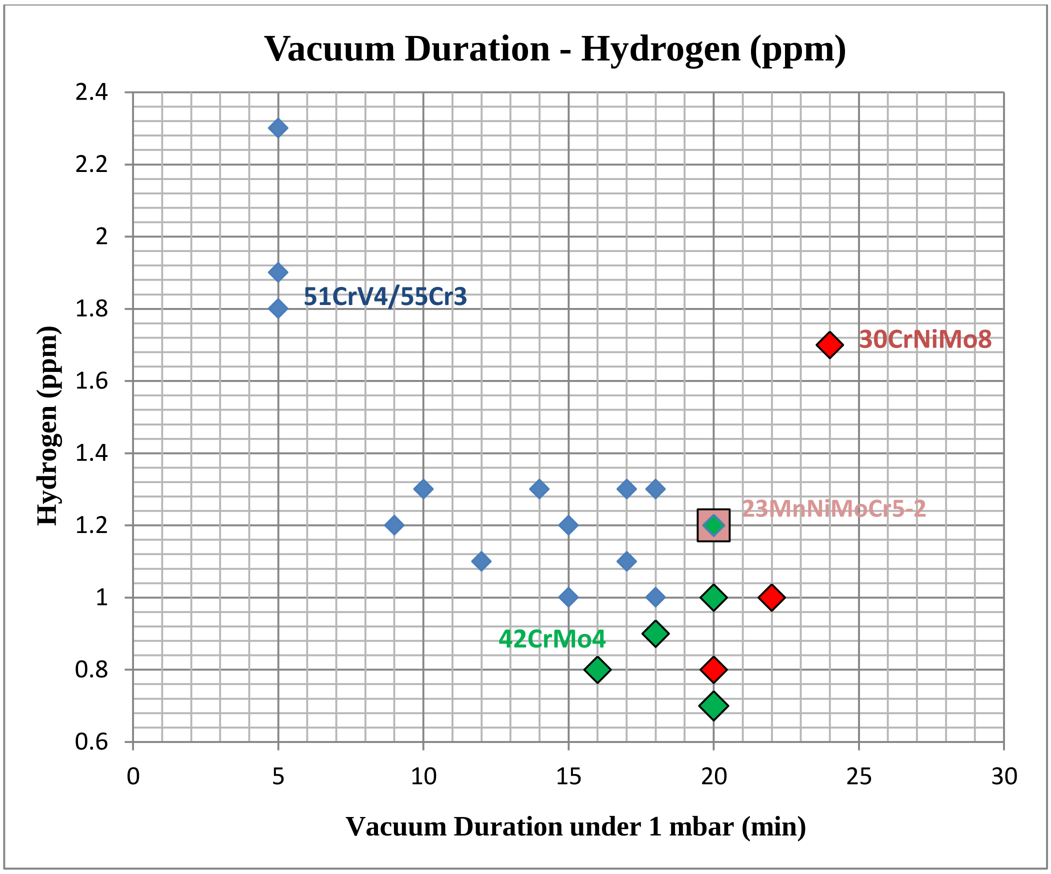


Figure 4.12 – Hydrogen contents of steel with respect to vacuum duration

Total oxygen contents of the heats given in Table 4.7 and other trial heats before and vacuum treatment were measured. Total oxygen contents, in ppm, of 30CrNiMo8/34CrNiMo6 steel grades, trial heats which were subjected to vacuum only once and those which were subjected to vacuum twice are given in Tables 4.8, 4.9 and 4.10, respectively. Heats subjected to vacuum are taken back to the ladle furnace where temperature of steel is increased to the desired level while Ar is passing through the metal; total oxygen contents of the trial heats at the end of this heating step are also listed in Tables 4.8 - 4.10 under the column "End of Refining". Total oxygen contents of some of the 30CrNiMo8/34CrNiMo6 steel grades were measured in the ladle furnace before vacuum and also in the tundish; these measurements are listed in Table 4.8 under the columns "Ladle Furnace", LF1 and LF2, and under the column "Tundish".

Table 4.8 – Total oxygen results at various stages of 30CrNiMo8/34CrNiMo6 steel grades (in ppm)

Steel Grade	**Heat No**	**Ladle Furnace**		**Before Vacuum**	**After Vacuum**	**End of Refining**	**Tundish**
		LF1	LF2	BV	AV	ER	T1
30CrNiMo8	2998	285	57	59	95	37	57
30CrNiMo8	2999	80	41	35	77	37	27
30CrNiMo8	3000	48		53	76	42	25
30CrNiMo8	3001	51		67	32	42	35
34CrNiMo6	3044	36		68	62	46	49
34CrNiMo6	3045	54		42	69	29	40
34CrNiMo6	3144	70		50	30	33	
34CrNiMo6	3145	43		37	23	21	

Table 4.9 – Total oxygen results belonging trial heats vacuumed once (in ppm)

Steel Grade	**Heat No**	**Before Vacuum**	**After Vacuum**	**End of Refining**	**Vacuum Duration**
		BV	AV	ER	
51CrV4	3361	62	36	25	14 min.
55Cr3	3515	96	60		5 min.
55Cr3	3516	58	39		5 min.
34CrNiMo6	3807	52	54		20 min.

Table 4.10 – Total oxygen results belonging to trial heats vacuumed twice (in ppm)

Steel Grade	Heat No	Before Vacuum	During Vacuum	After Vacuum	End of Refining	Vacuum Duration
		BV	DV	AV	ER	
51CrV4	3362	61	54	38	43	12+5 min.
51CrV4	3363	67	29	75	55	10+7 min.
51CrV4	3532	70	48	76		10+5 min.
55Cr3	3616	51	56	48		9+6 min.
42CrMo4	3731	39	53	60		18+2 min.
42CrMo4	3732	47	45	50		16+4 min.

The results presented in Tables 4.8 - 4.10 indicate that the total oxygen contents before vacuum are in the 40 - 90 ppm range and that total oxygen contents of some of the heats decrease while those of some other heats increase during vacuum treatment. This is clearly seen in Figure 4.13 where total oxygen contents of several heats before and after vacuum treatment are shown by different-colored lines joining the initial and the final total oxygen values.

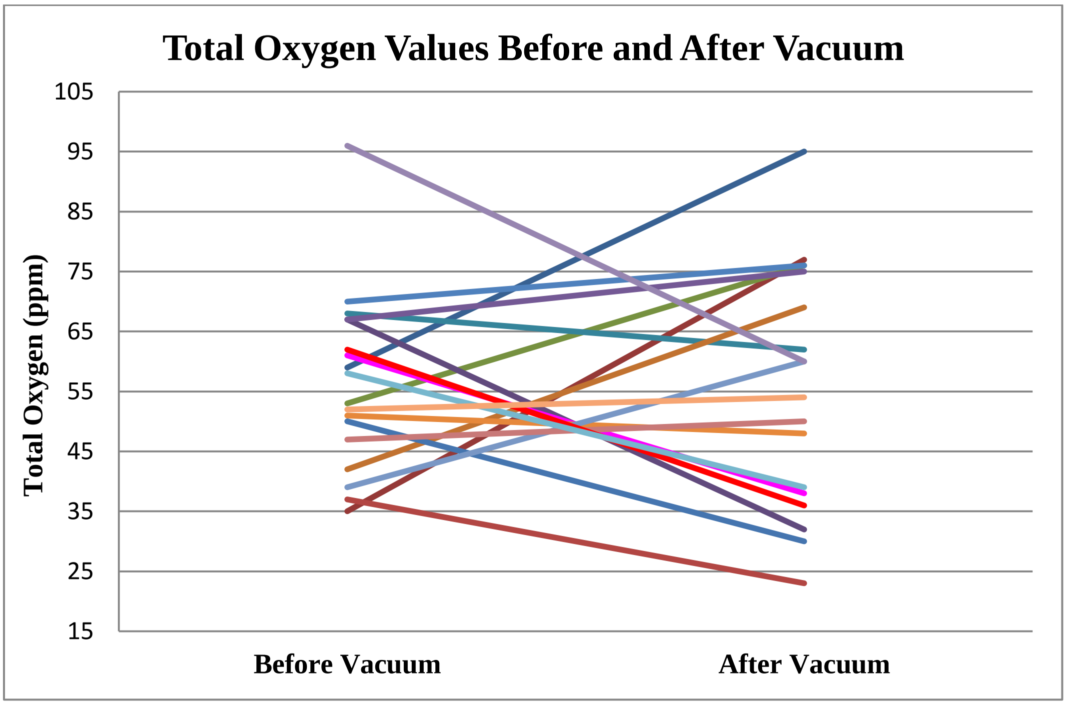


Figure 4.13 – Total oxygen values measured before and after vacuum

Duration of vacuum treatment is variable in Figure 4.13. Variation of the total oxygen contents of several heats with time in vacuum treatment is shown in Figure 4.14. Because total oxygen contents of these heats before vacuum are different, total oxygen contents of the heats are given as fractions of the initial total oxygen in Figure 4.14.

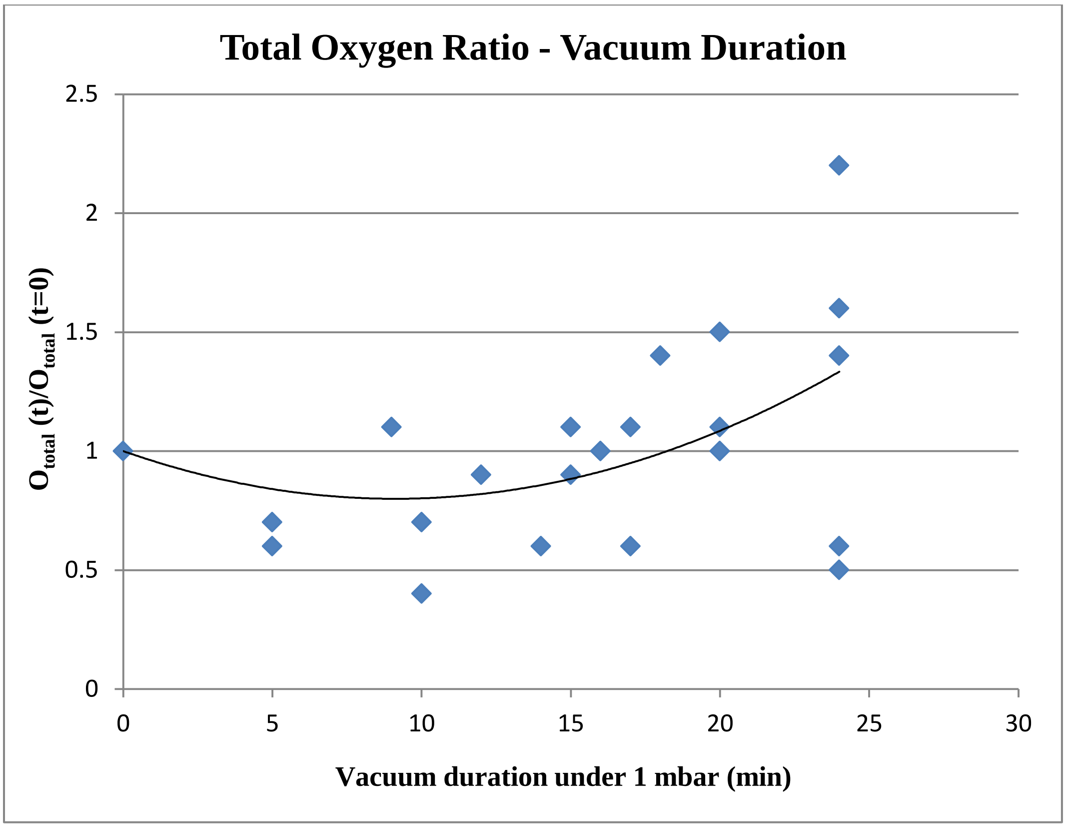


Figure 4.14 – Change in total oxygen values with respect to vacuum duration

Figure 4.14 indicates that total oxygen content of steel decreases in the first 10 to 15 minutes of vacuum treatment but increases for longer vacuum durations. There is about 50% decrease in the total oxygen contents of some of the heats up to about 15 minutes. $\frac{O_{total}(t)}{O_{total}(t=0)}$ ratio becomes larger than 1 for times longer than about 15 minutes indicating that there is reoxidation of steel after about 15 minutes. It was concluded previously, as stated above, that about 10 minutes of vacuum period is sufficient to decrease hydrogen to acceptable ranges. Based on these results, it is concluded that vacuum durations should be kept between 10-15 minutes and that times longer than 15 minutes should not be used at ÇEMTAŞ vacuum unit.

4.6 The Amount of CaSi Wire and Stirring Duration

As a major concern for clean steel, the amounts of CaSi wire used and argon stirring durations after vacuum were investigated for 66 trial heats. These heats were produced in the same campaign and rolled to the final products with equal diameters exposed to the same rolling reduction ratio. They are similar steel grades in accordance with their chemical composition so they were subjected to similar steelmaking parameters due to their composition.

For demonstrating statistical trial equality, the same numbers of heats were chosen as a trial for each steel grade. These similar steel grades are Grade A, Grade B and Grade C. The typical chemical composition intervals of these grades are given in the table below.

Table 4.11 – Chemical composition intervals of the trial heats

Grade A	***%C***	***%Si***	***%S***	***%P***	***%Mn***	***%Cr***	***%Al***
Min	0.17	0.25	0.000	0.000	1.40	0.10	0.020
Max	0.22	0.35	0.030	0.030	1.60	0.20	-
Grade B	***%C***	***%Si***	***%S***	***%P***	***%Mn***	***%Cr***	***%Al***
Min	0.17	0.00	0.000	0.000	1.10	1.00	0.016
Max	0.22	0.40	0.035	0.035	1.40	1.30	0.050
Grade C	***%C***	***%Si***	***%S***	***%P***	***%Mn***	***%Cr***	***%Al***
Min	0.17	0.00	0.020	0.000	1.10	1.00	0.020
Max	0.22	0.25	0.035	0.025	1.30	1.20	0.040

In Table 4.12, total holding time is the total time elapsed during stirring after vacuum and waiting in turret, and EMS is the average value of electromagnetic stirrer intensities for the first and the second strands.

Table 4.12 – Average steelmaking parameters for three different steel grades

Steel Grade	CaSi Wire Amount (meter)	Ar Stirring Duration after Vacuum (minutes)	Total Holding Time after Vacuum (minutes)	EMS (ampere)	Indexed Internal Defect	Indexed Production Loss
Grade A	51.56	20.75	27.81	80.00	208.14	250.02
Grade B	45.43	20.58	26.96	80.26	64.07	98.15
Grade C	25.00	24.72	33.52	80.29	13.86	24.79

The average values of several parameters for each steel grade are tabulated in Table 4.12. Moreover, Figure 4.15 shows the average values of electromagnetic stirring intensity for three different steel grades. It can be seen that the EMS intensities are approximately same for all three of the steel grades mentioned.

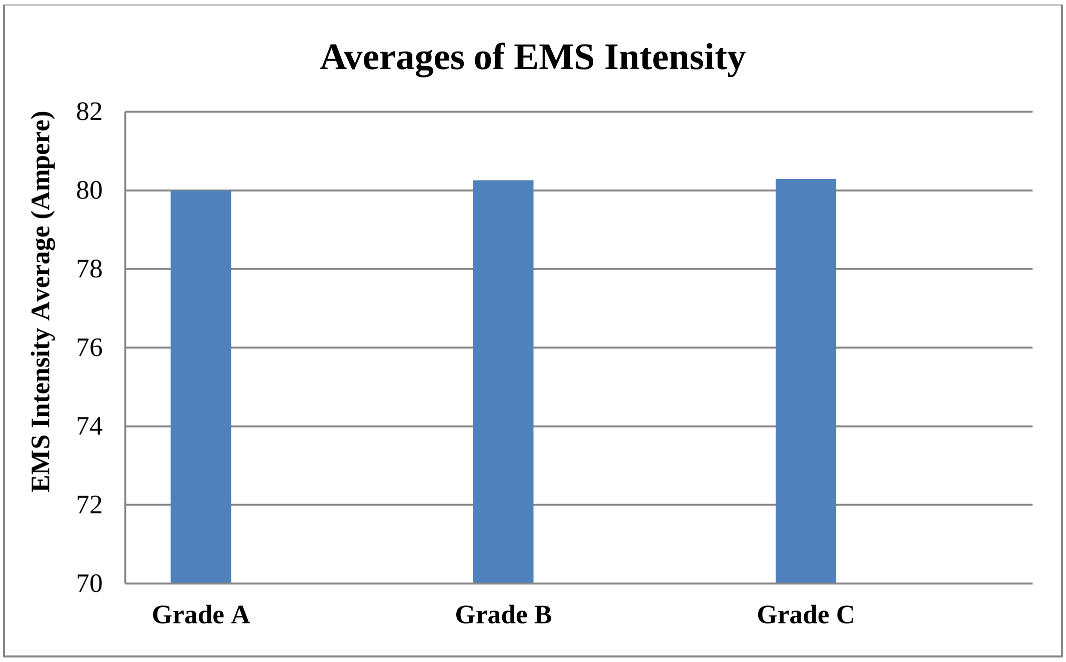


Figure 4.15 – Averages of EMS intensity for three different steel grades

Besides the average EMS intensities, the amounts of used CaSi wire for these grades are plotted below. Among the trial steel grades, the largest amount of CaSi wire was used in Grade A heats according to Figure 4.16. It was followed by Grade B and Grade C, although the aluminum contents of these three grades are almost at the same level.

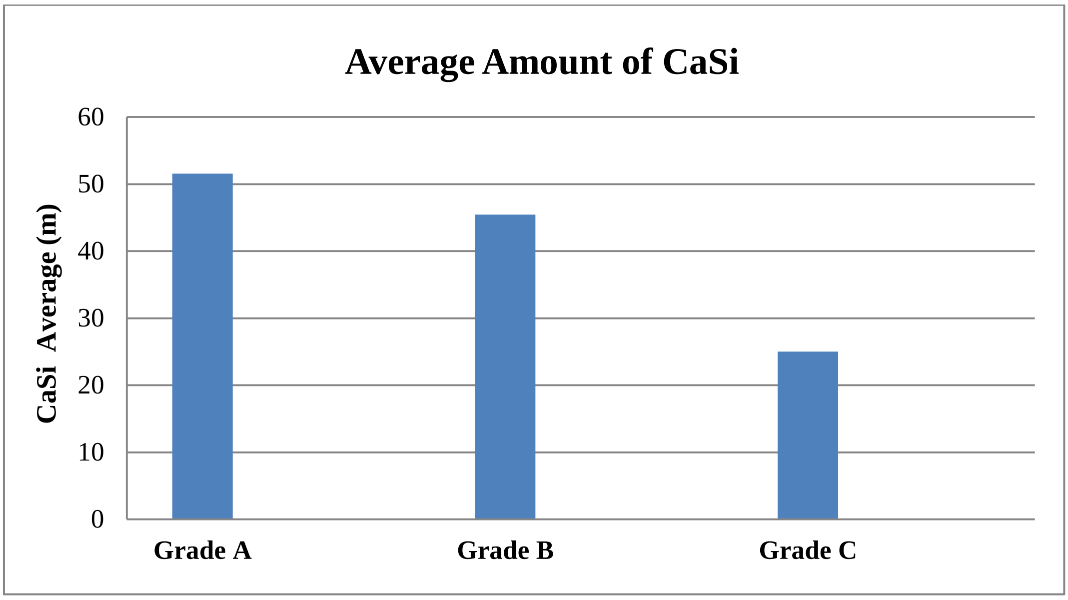


Figure 4.16 – Average amounts of CaSi wire used for the steel grades

In ÇEMTAŞ steelmaking practice, averages of argon stirring durations are shown with respect to steel grades in Figure 4.17. In this figure, the average of argon stirring durations for Grade C heats is nearly 4 minutes longer than the other two grades because this grade contains some amount of sulphur due to its typical composition interval and the sulphur wire is fed in to the steel after feeding CaSi wire. Hence, some certain time is obviously spent before the transportation of ladle to continuous casting. For Grade A and Grade B heats, there is no need to hold the ladle at the station so it is directly transferred to the turret. It is noticeable that the extra gentle stirring for 4 minutes affects the cleanliness of steel positively.

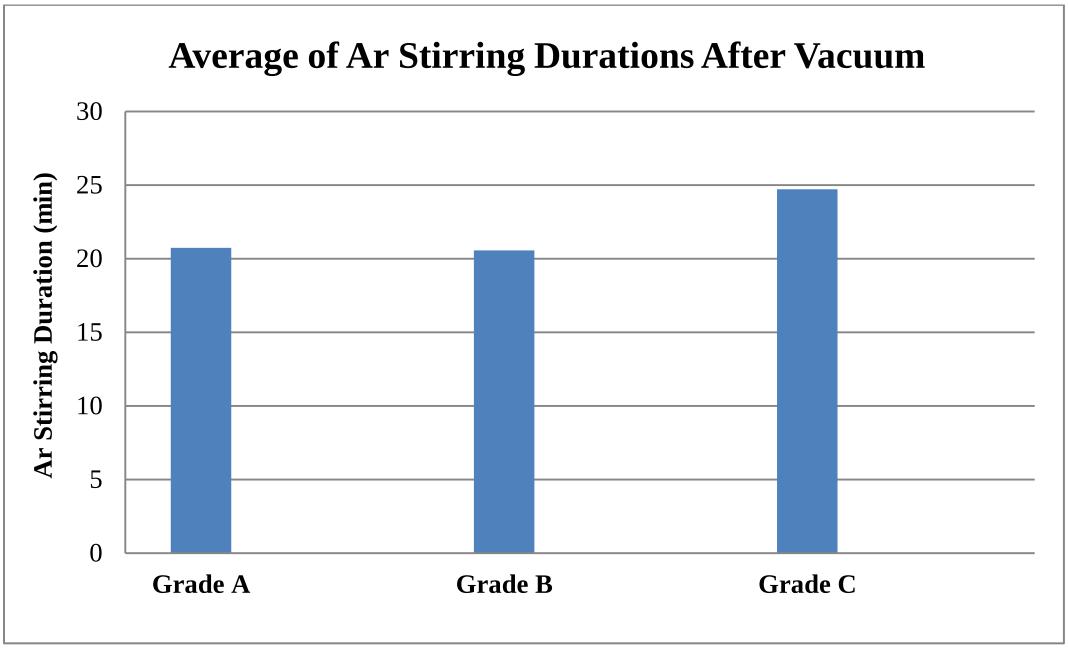


Figure 4.17 – Averages of argon stirring durations after vacuum

In Figure 4.18, average production losses, which can be accepted as measurement of steel cleanliness, are indicated with respect to the grades.

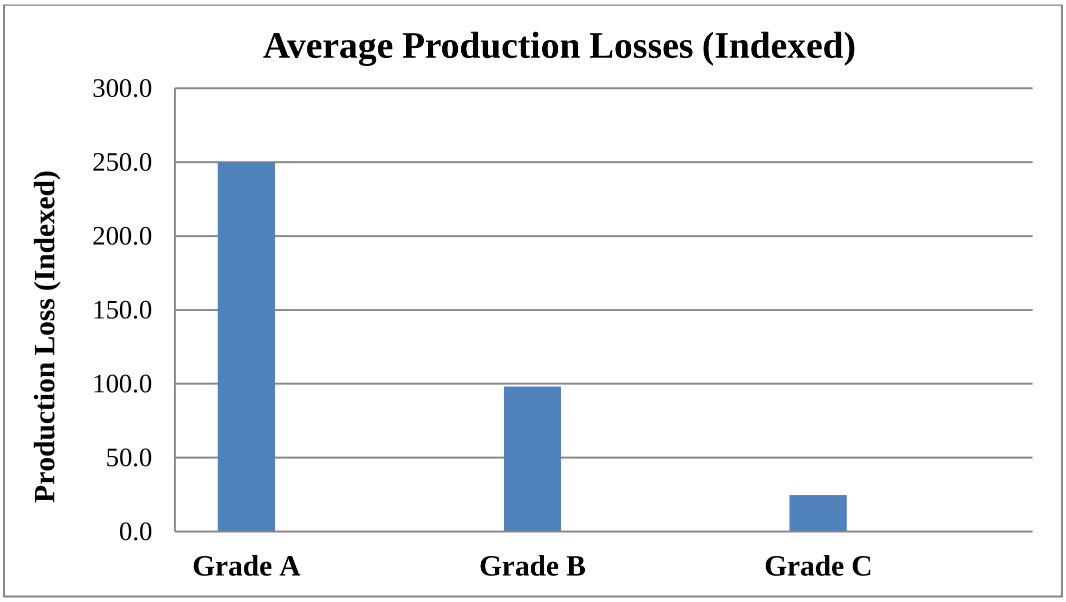


Figure 4.18 – Production losses belonging to trial heats

As it is observed from Figures 4.16 and 4.17, although the highest amount of CaSi wire is used, argon stirring duration is the shortest one for Grade A trial heats. On the other hand, although the amount of CaSi wire is less than the others the stirring time is longer for the Grade C trials.

This situation is well matched with the indexed production loss amounts of the three mentioned grades, which are given in Figure 4.19. According to this figure, it is favorable to feed more CaSi wire providing more stirring durations as regarding the steel cleanliness.

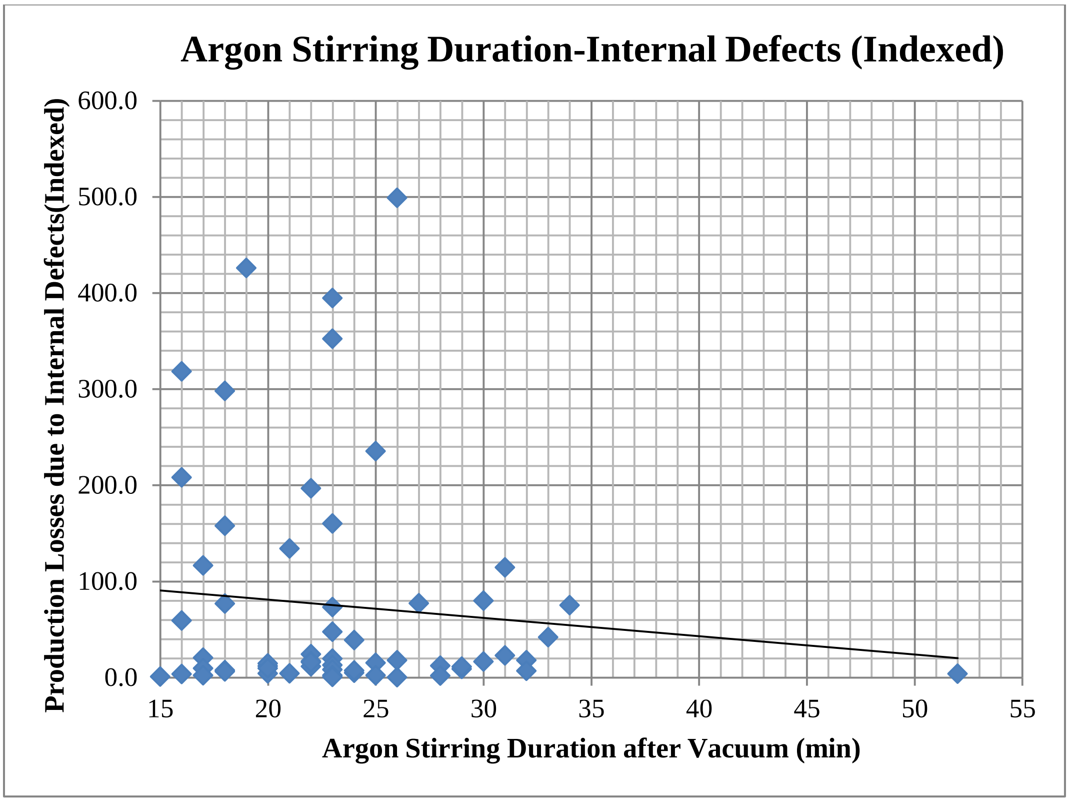


Figure 4.19 – Production losses due to internal defects (indexed) with respect to argon stirring durations

According to figure given above, change in production losses due to internal defects was investigated as a function of stirring durations after vacuum. It is apparent that there is a sharp decrease in the production loss after nearly 25 minutes stirring duration following the vacuum operation. Stirring the molten steel with argon about 25 minutes seems to be critical duration since the production loss amounts are higher for shorter times. In contrast, there is a tendency for production loss amount to decrease for longer times.

Due to the problems about lanced-opening and clogging, interferences are applied to 32 out of 66 heats with Grade A, Grade B, Grade C trials. Out of these 66, 14 heats are lanced opened and 11 of them are re-opened also using oxygen due to clogging of the nozzles after it was opened at the beginning of the casting. This situation is a sign of Al_2O_3 inclusions, which could not be transformed into liquid.

When the amount of the CaSi wire is appropriate, the Al_2O_3 inclusions present in the steel turn into liquid CaO-Al_2O_3 particles which would not cause nozzle blockage. Moreover, other inclusions would be globular by CaSi addition, which means that inclusion shape control is achieved. Consistent with these, Ca/Al ratio is a topic which is emphasized in literature and it is stated that the increase in this ratio would have positive effects on the cast and also on the cleanliness of the heat.

Samples taken from the end of refining stage are measured by ARL 4460 optical emission spectrometer. In order to observe the situation mentioned above, a correlation was made between the results of the spectrometer and non-destructive testing machines. According to Figure 4.20, it was concluded that when the $Al_{insoluble}/Ca$ ratio is smaller than 1 the production loss is lower and when this ratio increases and exceeds 1, scrapped amount of products increases as well.

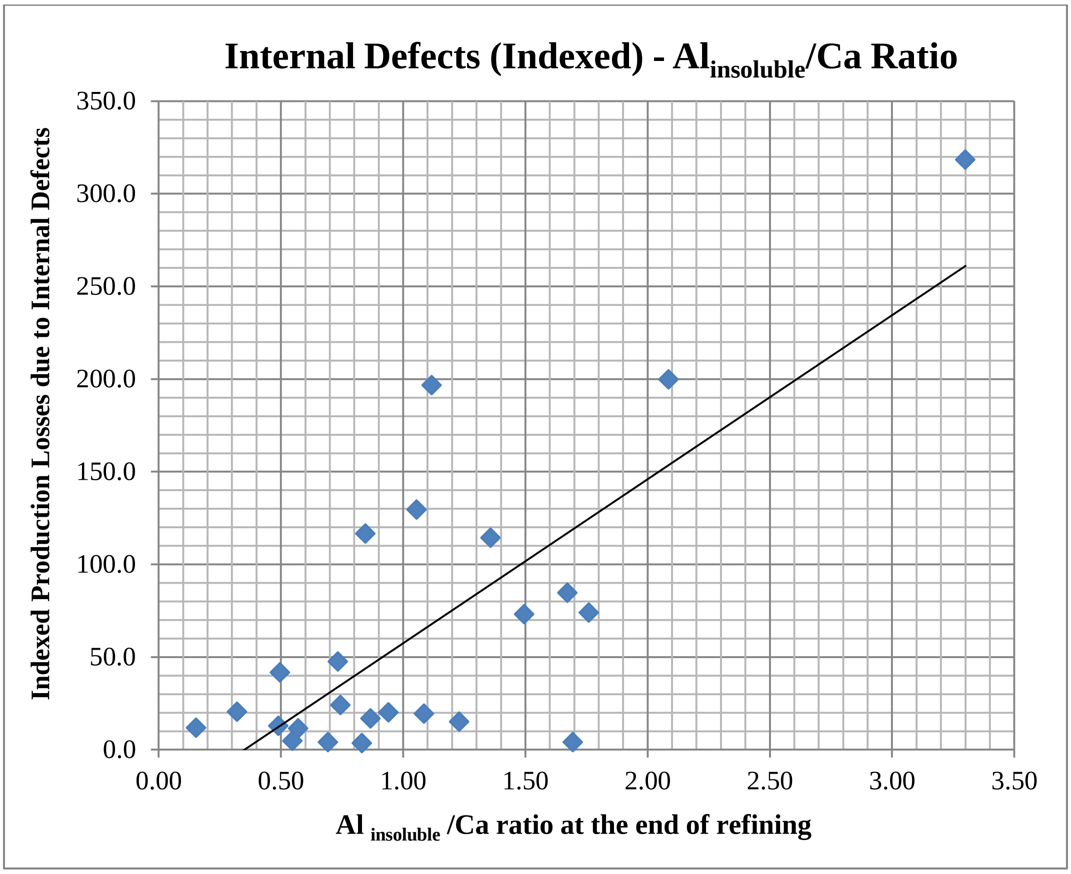


Figure 4.20 – Production losses due to internal defects with respect to $Al_{insoluble}$/Ca ratio

It is expected that for a constant Al amount, the increase in Ca in the steel would reduce the production loss amounts in ÇEMTAŞ steelmaking practice. In addition when $Al_{insoluble}$/Ca amount is 1, it corresponds to the 50%-50% weight percent in the binary Al_2O_3-CaO phase diagram.

Furthermore, based on Figures 4.15 to 4.18 investigating three types of grades, the Ar stirring at the end of vacuum and especially the stirring after Ca treatment affect the cast positively. Following the wire feeding, it is required that the intensity of the stirring should be low for clean steel production.

4.7 The Effect of Sulphur Content on the Steel Cleanliness

Sulphur is usually known with its adverse effects on the cleanliness and mechanical properties of steel. Moreover, the CaS which forms in Grade C heats affects the castability negatively owing to the sulphur content.

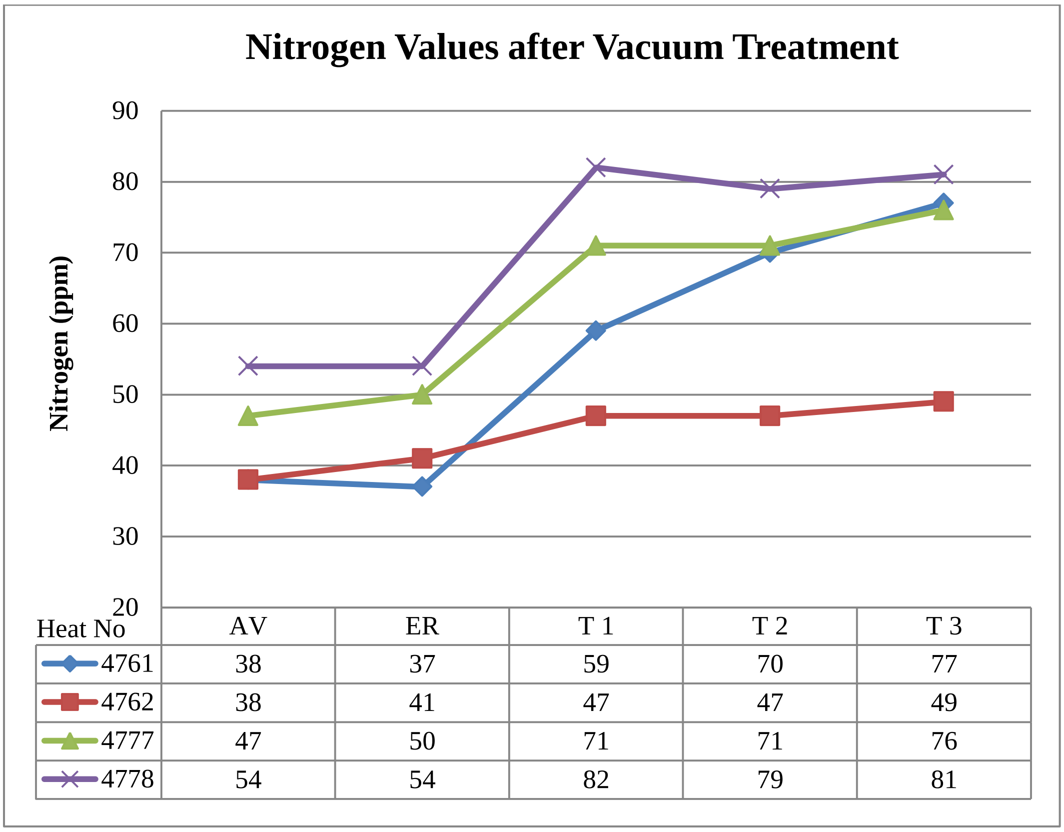
In contrast to this knowledge, Grade C grade has the least production loss among three trial grades, although it is the only grade that contains some amount of sulphur. The apparent distinction in production loss amounts between the heats containing sulphur and not containing sulphur lead to the investigation of differences between their production parameters. Furthermore, this circumstance is frequently valid at ÇEMTAŞ for the steel grade couples, one of which contains sulphur (0.020-0.035%S) and other contains trace amount (<0.005%S). The production loss amounts are observed and it has come out that the NDT results are lower for the heats containing sulphur. 20MnCr5-20MnCrS5, C45-C45R and 42CrMo4-42CrMoS4 are examples of these steel grade couples.

Although lower amounts of CaSi wire is used for Grade C, the reason for the lower amounts of the production losses can be explained by longer Ar stirring duration. The CaSi wire amount decreases with increase in sulphur content of steel since the sulphur in steel improves the calcium absorption of the bath as stated in literature review section.

4.8 The Usage of Ladle Shroud with Argon Purging System

During the production of trials with the heat numbers 4761, 4762, 4777 and 4778, samples were taken at the stages after vacuum treatment. Nitrogen contents of these samples were measured by LECO ONH 836 device.

The results of these measurements are shown in Figure 4.21. The samples taken from tundish at different times are numbered as T 1, 2 and 3, respectively.



Heat No	AV	ER	T 1	T 2	T 3
◆ 4761	38	37	59	70	77
■ 4762	38	41	47	47	49
▲ 4777	47	50	71	71	76
× 4778	54	54	82	79	81

Figure 4.21 – Nitrogen values at the stages after vacuum treatment for trial heats

According to this figure, it is notable that there is an obvious increase in nitrogen content of molten steel from the end of refining (ER) to the tundish (T1) for all trial heats. Based on this observation, some improvements were carried out so as to prevent nitrogen increase between the stages mentioned above.

As mentioned in Chapter 1, there are various methods for evaluation of steel cleanliness. One of them is nitrogen pick up of steel among indirect methods. It is widely used technique in steelmaking industry because of its rapid results and cost considerations. With this method, it is known that reoxidation of steel occurs when there is a positive difference between nitrogen contents of two different processes. Joints throughout the steelmaking process may lead to nitrogen increase, which means that reoxidation occurs at these joints.

In steelmaking industry, ladle shroud is a common tool which is attached to the ladle in order to protect steel stream flowing from the ladle to the tundish regarding on air entrainment. Moreover, the joint between the shroud and the ladle is also one of the probable origins of reoxidation. Argon purging system is installed to the shroud so as to prevent the oxidation of deoxidized steel.

With these purposes, numerous trials were carried out to determine the usage and the effectiveness of ladle shrouds with argon purging system. The heats with these shrouds are compared with the heats with ones without argon purging according to their nitrogen pick up.

Within the scope of these trials, the shrouds with argon purging were used in 190 trial heats that were produced in the day shifts. On the other hand, the ones without argon purging system were used in 118 trial heats that were produced in the night shifts. The trial heats were investigated concerning the nitrogen pick up, which is the difference the nitrogen content of the ladle at the turret and the nitrogen content of the steel in the tundish.

Table 4.13 – The averages of nitrogen pick up for two different types of shrouds

	Nitrogen Pick Up
The Shrouds without Argon Purging	16 ppm
The Shrouds with Argon Purging	9 ppm

According to Table 4.13, nitrogen pick up of steel was decreased from 16 ppm to 9 ppm by using the shroud with argon purging system. It is concluded that reoxidation can be prevented at these joints with the help of the shroud with argon system which acts as a shielding for atmosphere.

4.9 The Other Trials

In addition to the trials mentioned above, various parameters were examined with numerous trial heats with the objective of improving the steel cleanliness. After tapping of steel in to the ladle, two types of aluminum deoxidizers, which are different in composition and shape, were observed according to their efficiencies for deoxidation.

At the refining stage, aggressive argon stirring were applied to the molten steel in order to eliminate inclusions present in steel. Moreover, the chemical composition of slags was taken into account with regards to inclusion absorption ability of slags at the same stage. CaO/Al_2O_3 ratio of slag was tried to be kept between 1.7 and 1.8 to decrease melting point of slag and enhance the absorption ability. Regarding on the chemical composition, different kinds of tundish covering powder were used for the same ability. Furthermore, a special apparatus like pour pads were installed to the tundish so as to control the flow of steel in the tundish just after the opening of the ladle. Their efficiency was observed in many trials as well.

In continuous casting, intensity of electromagnetic stirrer was considered as variable and special gaskets were fitted into the joints between the submerged entry shroud (SES) and tundish nozzle. In some other trial heats, instead of SES, submerged entry nozzles (SEN) were used in tundish to examine the effect of reoxidation originated from the related joints. All variables mentioned in this section were particularly investigated in each trial heat and changes in production losses belonging to these heats were compared with the results of previous heats, which were produced by usual ÇEMTAŞ steelmaking practice. Most of the variables mentioned in Section 4.10 were found to be less effective for total oxygen values or production losses of the trials. Hence, it was concluded that they were not considered as the main sources of the defects examined in Section 4.1.

On the other hand, the variables like carbon content just before tapping, vacuum durations, argon stirring durations after vacuum and $Al_{insoluble}/Ca$ ratio were found to be effective for total oxygen values or production losses of the trials. Hence, it was concluded that the ratio of rejected bars to total production amount was decreased to 2.84% from 4.83%.

CHAPTER 5

CONCLUSION

This study was started with the purpose of examining the reasons of internal defects, which are detected by non-destructive testing machines, in the round bars produced at ÇEMTAŞ, taking precautions to prevent the formation of these defects and observing the effects of these precautions on the quantities of total oxygen content and production loss.

Macroscopic and microscopic examinations were conducted on the samples, which were taken from the faulty regions of the final products. Total oxygen measurements, chemical analysis, dissolved oxygen and hydrogen measurements from the molten steel were carried out during steelmaking processes. These examinations indicated that the cleanliness of steel plays an important role and the majority of defects originated from oxide inclusions. As a result of these measurements, it was focused on the investigation of possible sources of oxide inclusions. Hence, this thesis was directed to the stages of steelmaking, which were electric arc furnace, deoxidation, refining and continuous casting. Certain precautions were taken in order to produce cleaner steel. These precautions and their effects are given below:

- Inclusion content of steel should be as low as possible.
- Oxygen content of steel just before tapping was high; it was lowered by increasing the carbon content of the bath to as high values as possible in order to minimize the amount of deoxidation products. Thus, the carbon

content of molten steel should not be decreased to very low levels in electric arc furnace.

- Carbon boiling is required for flushing of hydrogen and nitrogen in molten steel, and also homogenization of the bath.
- High soluble oxygen contents after deoxidation in the ladle were found to be due to insufficient amount of aluminum addition during tapping. In order to decrease the soluble oxygen content just after deoxidation, the amount of aluminum to be added was found to be at least 1.6 times of the usual ÇEMTAŞ deoxidation practice.
- Extra aluminum addition should not be performed at the ladle furnace since it may lead to the formation of additional aluminum oxides.
- 9-15 minutes vacuum is enough to decrease hydrogen content to less than 1.5 ppm. Total oxygen content of steel decreases during the initial 10 to 12 minutes vacuum operation, but total oxygen content increases for longer vacuum durations.
- When the $Al_{insoluble}/Ca$ ratio is smaller than 1 the production loss amount is lower and when this ratio increases and exceeds 1 the production loss amount increases.
- When Al, Ca-Si and S wire is injected, liquid steel should be subjected to soft argon stirring for at least 3 minutes following the injection.
- After vacuum operation, stirring the molten steel with argon for about 25 minutes seems to be critical duration since the production loss amounts are higher for shorter times.
- The reoxidation of molten steel should be prevented and ladle shroud with argon purging should be used since nitrogen pick up of steel was decreased from 16 ppm to 9 ppm by using shrouds with argon system.

In the light of these findings, optimum production parameters were determined and applied for steel cleanliness. Reflections of the improvements were observed with respect to the total oxygen content of steel and production losses. Due to these precautions, the ratio of rejected bars was decreased to 2.84% from 4.83%. Furthermore, total oxygen contents of the final products were decreased to approximately 20 ppm levels from 30 ppm levels.

CHAPTER 6

FUTURE WORK

The present research has focused on the oxide inclusions regarding on the steel cleanliness. Although some hydrogen measurements were conducted after the vacuum treatment, any data for hydrogen does not exist at the following stages. Some of defects in the final round bars may be arisen from hydrogen in molten or solidifying steel. There are some difficulties to detect faulty regions originated from hydrogen because of high rolling reduction ratio of these bars. Furthermore, there is no specific test to detect these hydrogen defects in the rolled bars produced at ÇEMTAŞ. Thus, further studies should be directed to hydrogen control at ÇEMTAŞ Steel Plant.

Similar to hydrogen in steel, cooling regimes should also be investigated in continuous casting since capillary cracks, which may become larger during heat treatment, are difficult to be detected. Macroscopic examinations of billets and final products indicated that some transverse and also longitudinal cracks were observed in the vicinity of white band, which is formed as a result of electromagnetic stirring.

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