

# **Oxidic steel cleanness in high-carbon chromium bearing steel**

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## **OXIDIC STEEL CLEANNESS IN HIGH-CARBON CHROMIUM BEARING STEEL**

Ovako has an extensive R&D since many years, an area that now is in an even higher intensity. Some of the R&D work is published in our technical reports.

Due to that Ovako of today has had a number of different company names and used various trade marks we have until now chosen to not have these reports publicly available. However, many of these technical reports contain valid data about material and steel grades that we still promote, but with other names etc.

The following Technical Report from 1997 is part of a series of technical reports we issued in the late 1990's describing various aspects on how to achieve the clean steel we produce. These also show the development from earlier technical reports. See Ovako Archive Technical report 8/1983 and 1/1985, as well as 1/2000 about a later stage in this development.

Data and processes in this report represent state of art at time of publishing, and is to a large extent base for our current technology and capability.

In this Technical Report there is used the following Company names and trade marks that no longer is used by Ovako AB.

Ovako Steel; This company name is no longer used. The organization is now part of Ovako AB.

SKF; Is a separate company with no link to Ovako.

**Technical Report 1/1997**

**Oxidic steel cleanness  
in high-carbon chromium  
bearing steel**

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## **OVAKO STEEL**

Ovako Steel is the leading European producer of bearing steel and a major manufacturer of other special engineering steels. We are a fully owned subsidiary in the SKF group.

Our main strengths is in the field of long special engineering steel products – seamless tube, bar, heavy wire rod and surface removed wire. Rolled rings are also a specialty. A large part of the production is further processed by machining or forging. This share is increasing and illustrates our intention to progress towards further processing and higher specialization.

The most important customer segments are the rolling bearing industry, the automotive industry, and general machinery industries.

We have manufacturing units in Sweden, France, the U.K and the United States. Steel production is confined to Hofors, in Sweden. The production capacity is approx. 500,000 ton of special engineering steel per year.

### **Research & Development**

Our R&D mission is to pursue an efficient product and process development, adapted to existing and new technology, and within our product areas be recognized as the world leader in metallurgy, materials technology, machinability and metal cutting technology as well as heat treatment. The ultimate targets is to offer our customers the best total economy in their production.



## Abstract

The total oxygen content in the finished material has been used as a measure of steel cleanliness for high-carbon chromium bearing steel for many years. The average value has decreased on a yearly basis from 10 ppm in 1986 to 5 ppm in 1996. Even the deviation from the mean total oxygen value has decreased from 1.67 ppm in 1986 to 0.64 ppm in 1996, which indicates that the process is becoming more stable with respect to the oxidic inclusion content. Instead of focusing on the finished material, this study's objective has been to determine the degree of oxidic steel cleanliness of liquid steel under ladle treatment. Therefore, plant trials have been done where samples have been taken before and after vacuum degassing. The oxidic cleanliness has been determined by analysing the total oxygen content using melt extraction, the inclusion composition using a scanning electron microscope, and the inclusion size distribution using an optical light microscope following the swedish standard SS111116, (JK chart II).

**Key words:** *inclusions, cleanliness, ladle treatment.*

## Introduction

Non-metallic inclusions in steel have a great influence on the final properties of the finished bearing steel [1,2]. A specific example of this is the fatigue damage which is initiated under the raceway. Such fatigue problems originate in the loaded zone in weak areas of the material. The most serious source of these material defects are hard and brittle oxidic inclusions [3-5]. Another example is that the rolling contact fatigue life in bearing steel is also improved with a decrease in the total oxygen content [6]. Therefore, a top priority in the production of bearing steel is a reduction in the overall amount of oxidic inclusions and more effective methods of controlling the size, distribution, and composition of the inclusions that remain in the finished material.

The total oxygen content can be used as a first measure of the oxidic inclusion characteristics of the steel. It is possible to determine a relationship between total oxygen content and fatigue properties of bearing material. Fig. 1 shows a Weibull plot of rotating beam shot-peened samples for two bearing steels with different total oxygen contents. The specimen failures occur at smaller fatigue cycles for the material with a higher oxygen content: another example of how the fatigue properties can be improved by lowering the oxidic content of the bearing steel.

This paper describes our efforts to both minimize the total oxygen content of the produced roller-bearing material and more accurately determine the change in size, distribution and composition of oxidic inclusions during ladle treatment.

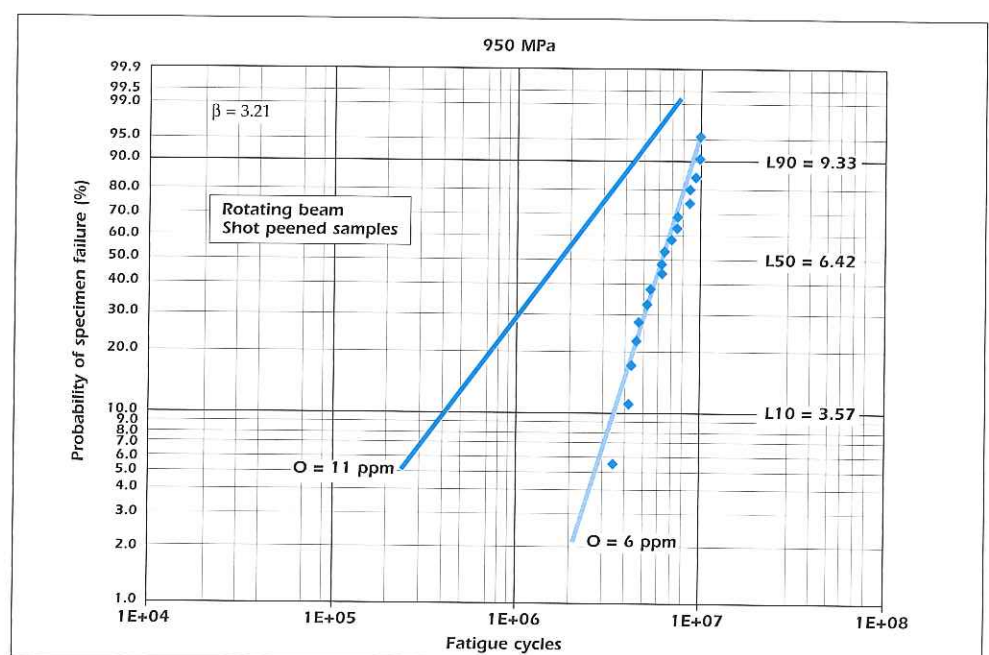


Fig 1. Probability of specimen failure as a function of fatigue cycles. Data for bearing steels at two different total oxygen contents: 6 ppm (G1578, Al=0.028%) and 11 ppm (V3343, Al = 0.035%).

## Plant description

A plant layout of the process route to for ingot production is shown in Fig. 2. Scrap is melted in a 100 t oval bottom tapped (OBT) arc furnace. Steel is tapped into a ladle and during this time predeoxidation takes place. After tapping of the steel into the ladle, careful deslagging of the furnace slag is done before transporting the ladle to the ASEA-SKF LF station. A synthetic slag is added in the LF followed by deoxidation and alloying. All steel is treated under vacuum. The LF is equipped with gas and induction stirring. After ladle treatment the steel is cast into ingots.

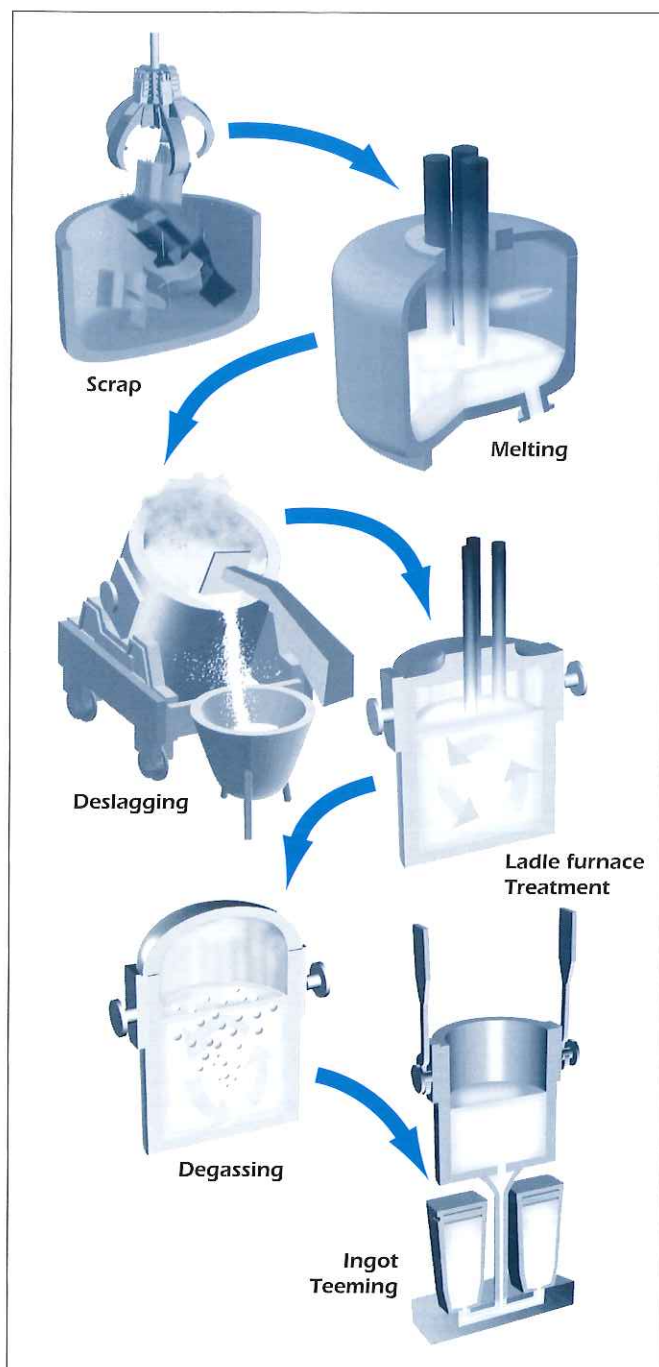


Fig. 2 Layout of the steel plant.

## Total oxygen content in the finished material

Many efforts have been made to decrease the total oxygen content of high-carbon chromium bearing steel in the last decade at Ovako Steel. As seen in Fig. 3, the total oxygen content for bearing steel has decreased from an average of 10 ppm in 1986 to 5.0 ppm in 1996. Important changes implemented in the process during this time period include: the usage of magnesite refractory in the ladles, deslagging before ladle treatment (1985 to 1987), the usage of a premixed synthetic top slag (1987 to 1989), and casting with an argon shroud (1992 to 1994).

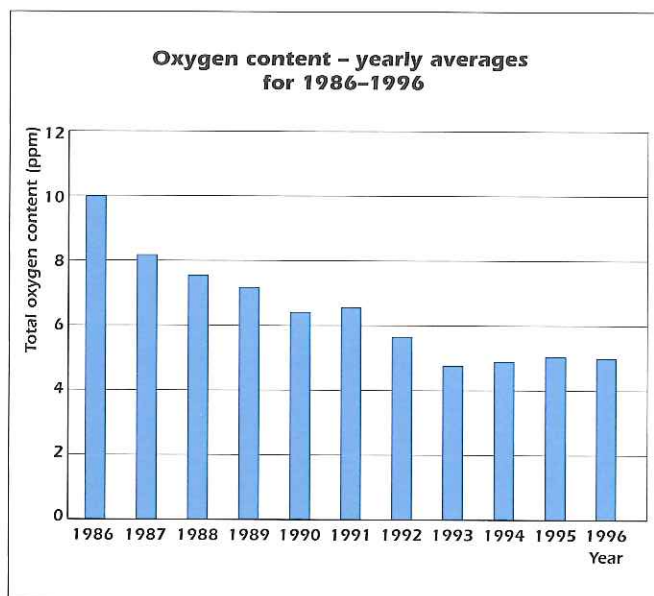


Fig. 3. Yearly average of total oxygen content in bearing steel during the last eleven years.

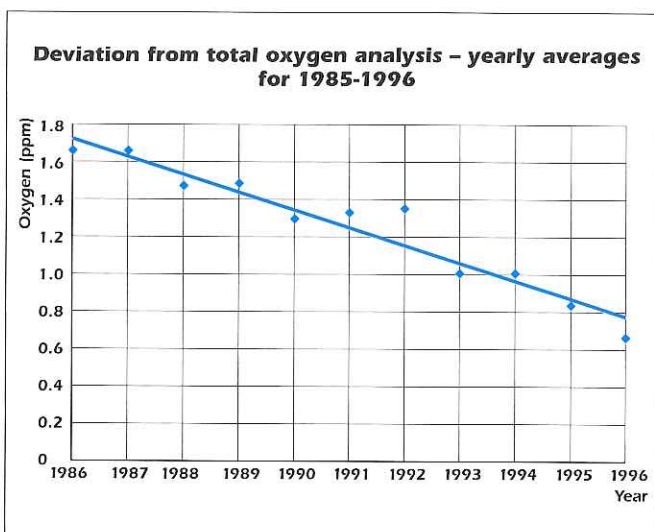


Fig. 4. Yearly average deviation from mean value of total oxygen content during the last eleven years.



Another important factor to consider is how the deviation of the total oxygen content from the mean value has changed during the same time period. As can be seen in Fig. 4, the deviation from the average total-oxygen value has decreased dramatically from 1.67 ppm in 1986 to 0.64 ppm in 1996. This decrease is almost continuous except for a small increase during 1991 and 1992. During this period there were problems with the exhaust system and the electrode regulation system in the ladle furnace, which might explain a larger deviation in the total oxygen content.

As seen in Fig. 3, the total oxygen content of the finished material during the last four years has remained almost constant at a value of 5 ppm. It seems unlikely that this value can be lowered much further. It is of course beneficial to decrease the deviation from the average value as much as possible since it gives an indication of the stability of the process with respect to the oxidic inclusion content.

So far only the oxidic inclusion content in the finished material has been the focus of most studies dealing with oxidic inclusions at Ovako Steel. We believe, however, that investigating the change in the oxidic inclusion characteristics during ladle treatment is also highly important, because at this stage of the process it is still possible to affect the final result.

## Changes in total oxygen content during ladle treatment

At first, the total oxygen content analysed from samples taken during ladle treatment was used as a measure of the inclusion characteristics, though it really only represented the total volume of oxidic inclusions. The approach was to perform some measurements of the total oxygen content during different stages of the ladle-treatment process. If the analyses from the liquid steel sampling seemed to vary somewhat, it was believed that these kind of samples could also be used to analyse composition and distribution of oxidic inclusions. In that way a more complete description of the inclusion picture could be obtained.

There were some questions as to what sampler to use, since earlier experiences with standard production dual thickness samplers had indicated that the deviation in analysis of the total oxygen content

was large. Different values of the total oxygen content were obtained when analysing different parts of the sample. Some of the possible reasons for this is that the sample may have contained pores which in turn may have contained oxygen, which would contaminate the sample. There is also a chance that oxidic inclusions had moved during solidification, which would cause the total oxygen content level to vary within the sample. The variation of total oxygen measurements within a cylindrical sample has also been pointed out by Marique et al [7], who showed that the deviation increased when a sample was taken from the upper part of the cylinder.

In a project sponsored by the Swedish Steel Producers' Association, researchers and technicians from MEFOS tested three different samplers in the LF [8]: dual thickness, TOS (Total Oxygen Sampler) [9], and TIC (Total Inclusion Control) [10] developed by MEFOS and Mekinor Metall. All samples were taken at the same depth and within 15 seconds. Significant reconstruction of a lance was necessary to make the TOS sampling automatic. Some typical results from these plant trials are shown in Fig. 5. Both samples taken by the dual thickness and TOS samplers were frequently not filled, and therefore homogenous and pore-free samples could not be obtained. This being the case, subsequent analysis of these samples was not carried out as resulting values would be highly unreliable. The TIC samplers rendered a more consistent sample quality which enabled us to obtain reliable values of total oxygen. Therefore, it was decided to use the TIC samplers for the continuing plant trials in evaluating the distribution and composition of oxidic inclusions.

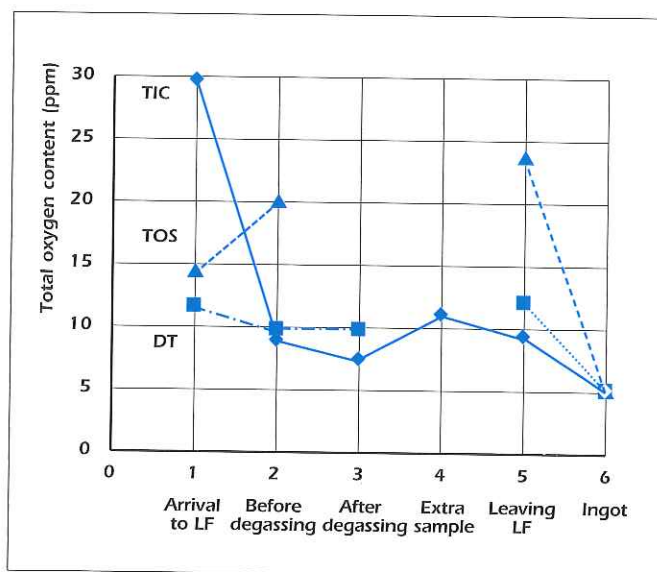


Fig. 5 Total oxygen content during ladle treatment analysed using three different samplers: TOS [9], dual thickness (DT) and TIC [10].



# Evaluation of inclusion characteristics during ladle treatment

## Experiments

Samples were taken using the TIC sampler during the following stages of the ladle-treatment process:

1. When the ladle arrives at the LF
2. After melting of synthetic top slag (control sample)
3. Before vacuum degassing
4. After vacuum degassing
5. When the ladle leaves the LF

Seven heats of high-carbon chromium bearing steel were studied. Similar results were obtained for all heats and therefore results for one representative heat are presented below.

## Analysis

The TIC samples were cut into 20 mm long pieces taken from the part of the sample that is filled last. From earlier investigations it was found that this part of the sample is most homogeneous and gives the most accurate analysis of the total oxygen content. The samples were cast in plastic in order to make it easier to polish them. The cast samples were sanded with a coarse sanding paper so that the center of the axial steel sample was reached, in order to achieve the maximum area for analysis. Finally, the samples were sanded with a fine sanding paper and polished with a diamond paste.

The size distribution of the inclusions was determined using an optical light microscope of 100x magnification. Inclusions above about 2.82  $\mu\text{m}$  were counted in a sample area of 60  $\text{mm}^2$  following the Swedish SS111116 standard, (JK chart II).

Inclusion composition was determined using a Scanning Electron Microscopy (SEM). In using an SEM, it is important to have a reliable electrical contact between the sample and the sample holder. Therefore, the samples were removed from the plastic cast form. The analysis was done using Kevex. Greater magnification was employed to detect inclusions in the range above 1  $\mu\text{m}$ .

## Inclusion composition

### sample 1: start of ladle treatment

A total number of 16 inclusions were detected. Eleven of these were pure  $\text{Al}_2\text{O}_3$  inclusions in the size range of 5.5 to 11  $\mu\text{m}$ , 4 were a mixture of  $\text{Al}_2\text{O}_3$

(16%),  $\text{SiO}_2$  and  $\text{CaO}$ , and one was a mixture of  $\text{Al}_2\text{O}_3$  (16%),  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$ . Probably the Fe, Si and Ca come from the added FeSi, since the amount is high and no other alloy is added before this particular sampling occasion.

### sample 2: after slag melting

A total of 9 inclusions were detected, seven of which were pure  $\text{Al}_2\text{O}_3$  inclusions within the size range of 1.5 to 5  $\mu\text{m}$ . One rather large inclusion (40  $\mu\text{m}$ ) contained a mixture of  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{MnO}$  and  $\text{TiO}$ . Another large inclusion of size 65  $\mu\text{m}$  contained a mixture of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ . The source of both these large inclusions is probably FeSi and aluminium wire.

### sample 3: before vacuum degassing

Ten inclusions were analysed, six of which were composed of pure  $\text{Al}_2\text{O}_3$  within the size range of 1.5 to 4  $\mu\text{m}$ . One inclusion with the size 6  $\mu\text{m}$  was composed of a mixture of  $\text{SiO}_2$  and  $\text{MgO}$ . The high  $\text{SiO}_2$  content indicates that the inclusion originates from FeSi. One 7  $\mu\text{m}$  inclusion was composed of a mixture of  $\text{Al}_2\text{O}_3$  (13%) and  $\text{CaO}$  (87%). The calcium most likely originated from FeSi, since the amount is so high. Two of the inclusions were 2.5  $\mu\text{m}$  and composed of  $\text{Al}_2\text{O}_3$  and  $\text{MgO}$  (10%). The magnesium presumably originated from an alloy, from top slag stuck on the lining from the previous heat, or from the lining.

### sample 4: after vacuum degassing

Ten inclusions were found, of which only one was pure  $\text{Al}_2\text{O}_3$  of size 2  $\mu\text{m}$ . The other nine inclusions in the size range of 1 to 8  $\mu\text{m}$  were composed of a mixture of about 80%  $\text{Al}_2\text{O}_3$  and 20%  $\text{MgO}$ . According to Hallberg [11],  $\text{MgO}$  is reduced from the lining by carbon during vacuum degassing and magnesium gas enters the steel and combines with  $\text{Al}_2\text{O}_3$  inclusions to form a complex magnesia-aluminate inclusion.

### sample 5: end of ladle treatment

Four inclusions were found of which three in the size range of 1.5 to 3  $\mu\text{m}$  were composed of about 80%  $\text{Al}_2\text{O}_3$  and 20%  $\text{MgO}$ . One inclusion also contained  $\text{CaO}$ .



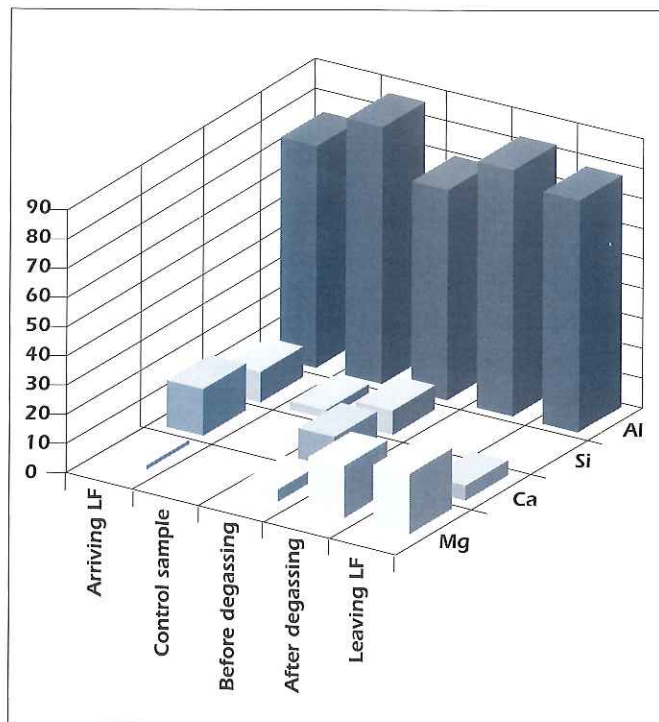


Fig. 6. Change in inclusion composition during ladle treatment

The change of inclusion composition during ladle treatment is summarised in Fig. 6. In the beginning of the ladle treatment, inclusions contain the deoxidation product  $\text{Al}_2\text{O}_3$  and other oxides such as  $\text{SiO}_2$ ,  $\text{CaO}$  and  $\text{Fe}_2\text{O}_3$ . The latter oxides probably originate from the alloy  $\text{FeSi}$  and furnace slag. During the later stage of the process none of these complex multicomponent oxide inclusions can be found. Before vacuum degassing the majority of the inclusions are alumina inclusions. After vacuum degassing the alumina inclusions have received a relatively high content of magnesia. A typical SEM picture of such a magnesia-alumina oxide inclusions is shown in Fig. 7.

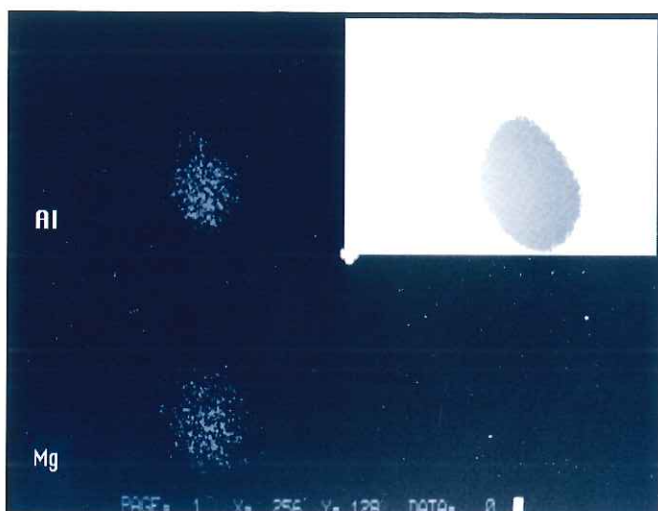


Fig. 7. SEM analysis of a sample taken after vacuum degassing.

### Inclusion distribution

The total oxygen content decreases throughout the ladle treatment process (except for a slight increase after vacuum degassing), as shown in Fig. 8. This also means that the number of inclusions also decreases, as illustrated in Fig. 9. This figure illustrates a tendency for the amount of large inclusions to decrease from the beginning of ladle treatment to the end of ladle treatment. A gradual reduction in the number of large inclusions has also been reported by Feng [12].

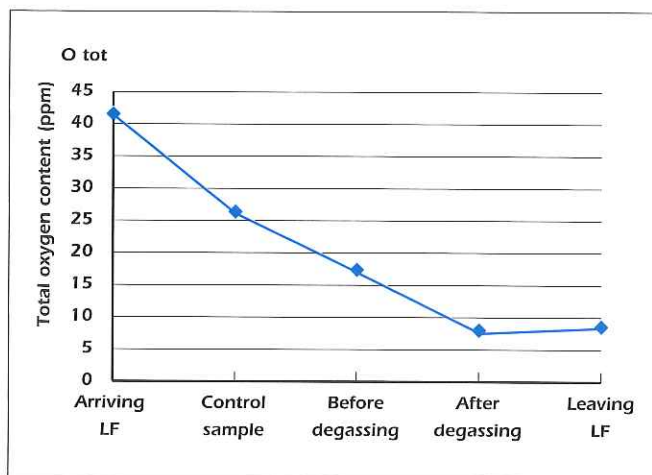


Fig. 8. Change in total oxygen content during ladle treatment.

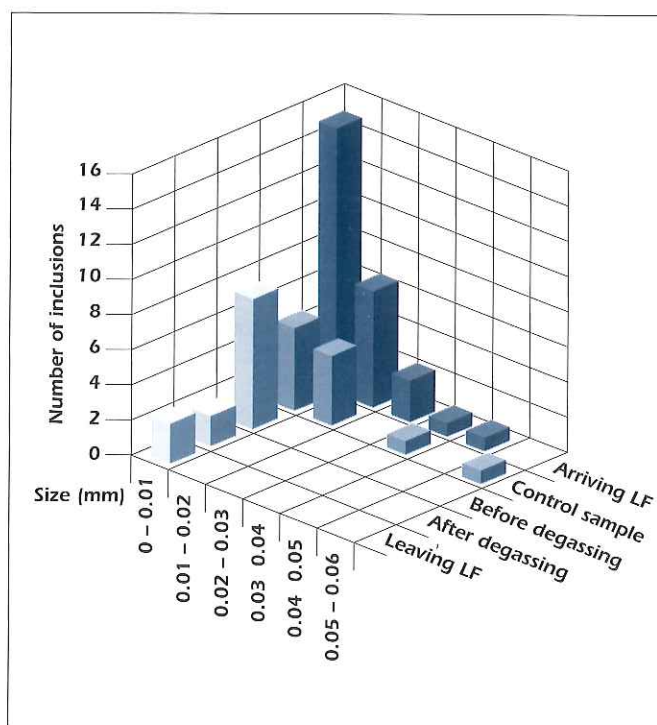


Fig. 9. Change in size distribution during ladle treatment.

## Discussion

The many efforts made at Ovako Steel during the last decade to decrease the oxidic content in high-carbon chromium bearing steel have been successful. The yearly average total oxygen value from analyses of the finished material has decreased from 10 ppm in 1986 to 5 ppm in 1993. Since 1993 the total oxygen value has been stable at this value. However, the deviation from the average total oxygen value taken on a yearly basis has decreased over the last three years. This indicates that the stability of the process with respect to the total oxygen content in the steel is still improving.

Further work on studying the oxidic cleanness during ladle treatment has been performed. It has been found that the TIC samplers give the most reliable total oxygen analysis for our purposes. The total oxygen content, which is a measure of the total amount of the oxidic inclusions, has been determined to decrease steadily during ladle treatment. The composition of the oxidic inclusions in the beginning of ladle treatment is mainly pure alumina resulting from deoxidation with aluminium. Some inclusions also contain silica, calcium oxide and iron oxide, which probably results from the FeSi alloy added during tapping or furnace slag. Before vacuum degassing the inclusions mainly consist of pure alumina. After degassing the alumina inclusions are transformed into a mixture of alumina and magnesia. During vacuum degassing magnesia is most likely picked up from the refractory and reacts with the alumina inclusions.

Inclusions ranging up to 60  $\mu\text{m}$  are present in the steel before vacuum degassing. After vacuum treatment, larger inclusions have been separated from the steel melt and mainly inclusions smaller than 10  $\mu\text{m}$  remain in the melt.

Future investigation of the oxidic content of liquid steel during ladle treatment will concentrate on:

- more detailed assessment of the effects of deoxidation on the resulting inclusion characteristics in the ladle before vacuum degassing
- the effects of stirring conditions after vacuum degassing on the composition and size distribution of the inclusions
- the effects of composition change in complex  $\text{Al}_2\text{O}_3\text{-MgO}$  inclusions on mechanical properties such as fatigue properties

- detailed assessment of the macro-inclusion content during ladle treatment and casting using specific sampling techniques followed by ultrasonic testing

Finally, it should be stressed that it is also essential to continue working on improving reproducibility of the process with respect to oxidic content. In other words, it is essential to make each heat in a similar way as the previous one. Here, the deviation from the total oxygen content can serve as a good measure of the degree of success.

## Acknowledgements

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