Inclusion Characteristics in Bearing Steel Before and During Ingot Casting

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ABSTRACT

Size, distribution, and compositions of inclusions were studied during secondary refining and casting in application to high-carbon chromium bearing steel production. Samples were collected in the ladle, tapping stream and mold. Total oxygen content, number of inclusions and composition of inclusions were then determined using melt extraction, optical microscopy and scanning electron microscopy, respectively. The results show that the inclusion characteristics do not change significantly during the final treatment in the ladle. A future change in stirring conditions may therefore be necessary. The analyses of the samples taken in the tapping stream and in the mold showed an increase in the oxide inclusion content. This increase is most likely due to reoxidation during sampling.

1. INTRODUCTION

Material defects have a large influence on properties such as fatigue strength in finished bearing steel. Most often, the source of the fatigue problems are hard and brittle oxide inclusions /1/. Larger inclusions have a more negative effect on the fatigue life than smaller ones /2-4/. In general, rolling contact fatigue life improves as the total oxygen content - both oxygen in the inclusions and dissolved oxygen - decreases. It is important to lower the dissolved oxygen content, due to the formation of inclusions as a cooling steel's capacity for dissolved oxygen decreases. Bearing steel production, therefore, attempts both to lower total oxygen content, and to control size, distribution, and composition of the inclusions which remain in the final product.

During deoxidation, an added deoxidant such as aluminum reacts rapidly with the dissolved oxygen through the nucleation of small alumina inclusions. The dissolved oxygen quickly lowers to a new equilibrium value determined by both the deoxidant and the temperature. The oxides tend to then increase in size from collisions caused by velocity gradients. A buoyancy force, initiated by density differences between the oxides and the steel and by the movement of small eddies in the turbulent flow, enhances the separation of the oxides from the steel into the slag. Upwards stirring also increases the separation of the inclusions, by causing them to collide and by lifting them up to the top slag where they can separate and assimilate.

From 1986 to 1996, the total oxygen content in OVAKO's high-carbon chromium-bearing steel has decreased from 10 ppm to 5 ppm (Figure 1). Many efforts are still spent on more reliably controlling inclusion characteristics. Although a lot of information is available on the final oxide content, not much is known about oxide content variance during production. Three previous studies have been carried out at OVAKO Steel on how inclusion characteristics change during different parts of the steelmaking process /5-7/. First, a general investigation was done to evaluate how the number and composition of inclusions change from the beginning to the end of ladle treatment /5/. The second study focused on the effect that deoxidation during secondary refining has on inclusion characteristics /6/. The third study determined the effect of final induction stirring (at the end of secondary refining) on inclusion characteristics /7/.

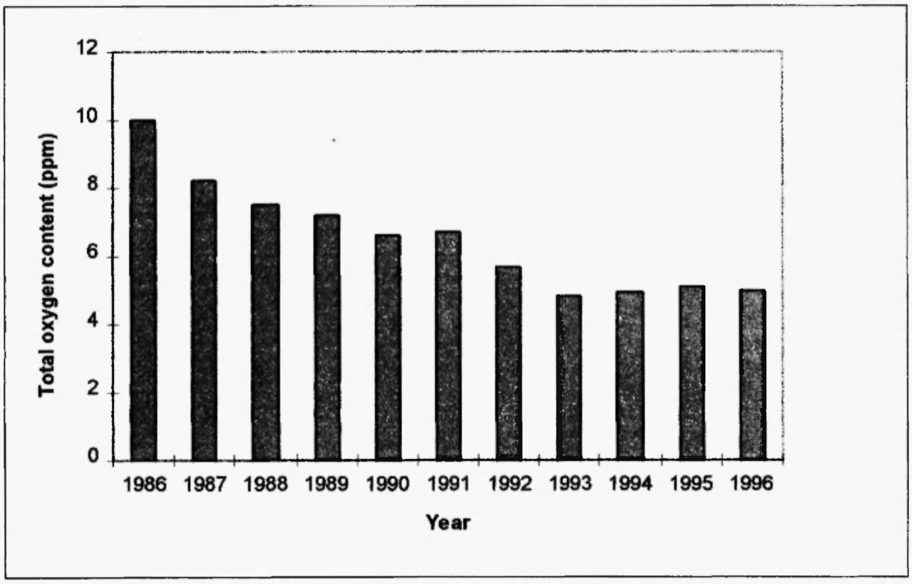

Fig. 1: Average yearly total oxygen content for high-carbon chromium-bearing steel from 1986 to 1996.

The present work is a continuation of these earlier studies /5-7/. It focuses on inclusion characteristics before (in the period at the end of secondary refining) and during casting. The paper describes the steel-making process at OVAKO, the on-line samplers used, and the experimental procedure. The results are presented and their influence on the process is discussed.

2. PLANT TRIALS

Plant Description

Ovako Steel produces scrap-based steel (Figure 2). The scrap is melted in a 100 t oval bottom-tapped (OBT) arc furnace. After adjusting the steel to the desired phosphorous, carbon, and temperature levels; the steel is tapped into a ladle while undergoing pre-deoxidation. It is then deslagged before being transported to the ASEA-SKF ladle furnace (LF) station. Once at the LF station, a synthetic slag is added. The secondary refining process which follows consists of three main steps. First, induction stirring is used during the alloying, deoxidation, and melting of the synthetic top slag. Second, gas stirring enhances the vacuum degassing operation, where hydrogen and sulfur refining are done. Thirdly, induction stirring is again used after the vacuum degassing to further promote the separation of inclusions from the steel to the top slag. During this phase the steel and slag are also heated with the electrodes to achieve the right casting temperature and to promote assimilation of inclusions. Upon completion of ladle treatment, the steel is tapped through an open stream surrounded by argon gas into uphill teeming trumpets. As the steel rises in the ingot mold, an insulate powder, which also helps prevent reoxidation, covers its surface. The exothermic cap of the ingot mold acts as a second means of maintaining a more homogeneous composition in the ingot. A single ladle fills three, eight-mold casting planes.

Sampling

In twelve production heats of high-carbon chromium bearing steel, samples were taken at four different stages during the production: after vacuum degassing (A), at the end of the ladle treatment (B), in the tapping stream after casting one third of the steel (C), and in the mold after casting two thirds of the steel (D).

Together with MEFOS /8/ and Electro-Nite Rescon /9/, a 35 mm diameter round sampler was developed with a 6 mm thickness and a 6 mm inlet quartz tube. The sampler design follows two main criteria: i.) the steel should solidify at a rate fast enough that the

Fig. 2: Outline of Ovako Steel's scrap-based process for production of ingots.

inclusions do not move during solidification (this is a problem for thicker samples /6/), and ii.) the sample should be easy to prepare for analysis using the existing equipment. The rapid solidifying (RS) sampler was used during sampling in the ladle and in the tapping stream; however, it could not be used in the mold since it did not fill satisfactorily. Instead, a round, dual-thickness (8 mm diameter inlet and 16 mm diameter body) vacuum tube (VT) sampler from Mekinor Metall /10/ was used for mold sampling. This sampler has been found to give the most homogeneous mold samples for our steel grades.

Both after vacuum degassing (A) and after ladle treatment (B), the RS immersion sample was taken using the existing automatic sampling equipment at the ASEA-SKF ladle furnace. This sampling system is pre-set to sample at a depth of 300 mm below the slag/metal interface. The rather large immersion depth is used to minimize the risk of sampling any top slag that is mixed into the steel while immersing the sampler. The RS sample was used in a stream-type design when sampling in the tapping stream (C). During this manual sampling, the quartz tube was kept at approximately 45o to the tapping stream, as recommended by the supplier /9/. The filling of the sample, due to the force of gravity and the cohesion of the tapping stream, was significantly more forceful than in the A and B samples. The VT sampler was used to sample in the mold. Just after the filling of the second casting plane, the sample was taken manually by immersing the sampler through the mold flux and into the steel at a depth well below the steel/flux interface. The sample was taken at some distance from the interface to minimize the risk of flux getting entrapped into the sample.

3. ANALYSIS PROCEDURE

After cooling, the samples were cast in plastic for easier polishing. The surface was first treated with course sanding paper, then with fine sanding paper, and finally polished with diamond paste.

Total Oxygen Content

The total oxygen content was determined using the melt extraction technique. The analysis procedure, done using Strohlein On-MAT 8500 equipment, is partially robotized in order to minimize the influence of human error on the results.

Inclusion Size Distribution

The size distribution was determined under 200x magnification using optical light microscopy. Magnifications of 500x and 1000x were occasionally used for better identification. On each sample, inclusions were counted within a 160 mm^2 area and classified according to the Swedish standard SS111116 (JK chart II). Since the inclusions found in liquid steel samples are in general not deformed, all were classified as D types. Light (DT), medium (DM), heavy (DH), and particular (DP) inclusions were counted according to the following size ranges: 2.8 $\mu m < DT < 5.7\ \mu m$, 5.7 $\mu m < DM < 11.3\ \mu m$, 11.3 $\mu m < DH < 22.6\ \mu m$, and $DP > 22.6\ \mu m$. The number of inclusions per mm^2 was then determined using the PCMIC software program.

Inclusion Composition

Scanning electron microscopy (SEM) was used to analyze the composition of the inclusions. The composition was determined using an energy dispersive technique. Samples were covered in a thin layer of gold due to the plastic surrounding the sample. When scanning the sample, the first 20 inclusions found above 2.0 μm were selected. A composition chart and the oxide percentages were recorded for each inclusion.

4. RESULTS

The samples taken in the mold were found to be very porous during examination with an optical microscope, even though they looked homogeneous from the outside. Therefore, the number of inclusions and the composition of the inclusions were not determined from these samples.

Total Oxygen Content

Due to the extremely low oxygen content of OVAKO's high-carbon chromium ball bearing steel, accurate measurement of total oxygen content requires a homogeneous sample, careful sample preparation, and a reliable analysis procedure. In order to analyze the most homogenous part of the sample, the cylindrical inlet, which has a smaller diameter, was used in the analyses of the total oxygen content. For the two measurements made of each sample, often one or both of the recorded total oxygen values were extremely high. During sampling, pores formed which were not previously present in the steel. These pores can be caused by the interface between the liquid steel and the initial air in the sampler, by the highly turbulent convection flow mixing this air into the liquid steel, and by an insufficient pressure being retained in general. During determination of the total oxygen content, the oxygen in these pores is included in the total oxygen value. According to the experience at OVAKO, the likelihood of a total oxygen content dropping from above 11 ppm at the end of the secondary refining operation to the average 5 ppm of the finished steel is extremely slim. All total oxygen values above 11 ppm were therefore removed from the data analysis. For the samples taken in the tapping stream and in the mold, the total oxygen contents were very high. For the tapping stream samples, only 4 analyses were lower than 11 ppm. Therefore, the total oxygen data for the tapping stream samples was excluded from the comparison. The samples from the mold were also quite porous and therefore many analyses were above 11 ppm.

The data from the same samples as those used in the inclusion size distribution data were then averaged and graphed. The average data for the total oxygen content in finished rolled bars for all heats of high-carbon bearing steel produced during 1997 were also included. As can be seen in Figure 3, the total oxygen content goes down slightly after induction stirring, increases in the ingot mold, and then decreases to below any of the former values by the finished rolled bar.

Inclusion Size Distribution

The variation of the average total number of inclusions from the start of induction stirring to the finished bars is shown in Figure 4. The number of inclusions only changed slightly during induction stirring, but was higher in the tapping stream. By the time the steel reached the finished rolled bar stage, the total number of inclusions fell below the number of inclusions found in the first sample. When the inclusions were broken down by size (Figures 5 to 7), this same pattern was found in the averages of the total

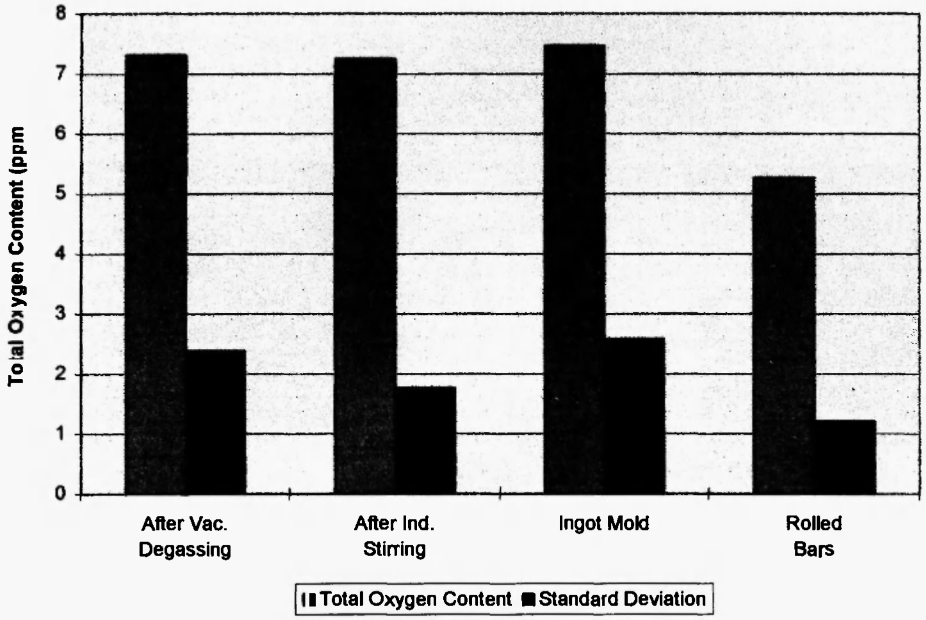

Fig. 3: Variation in total oxygen content from end of vacuum degassing to rolling.

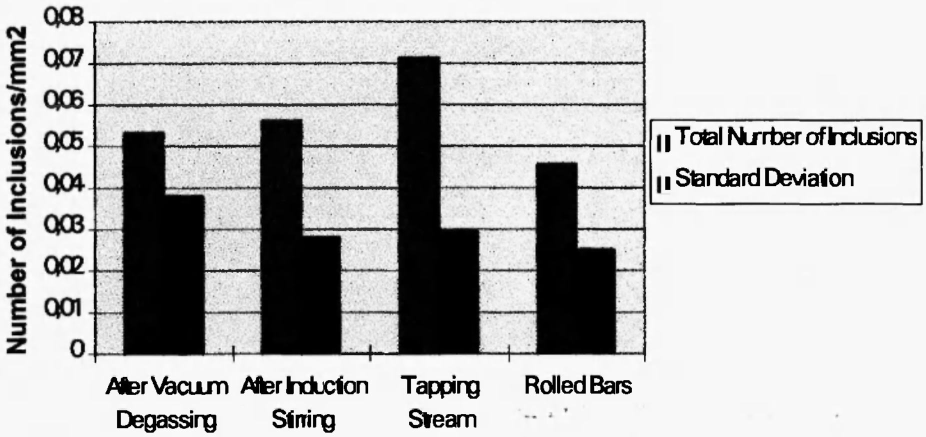

Fig. 4: Variation in total number of inclusions from end of vacuum degassing to rolling.

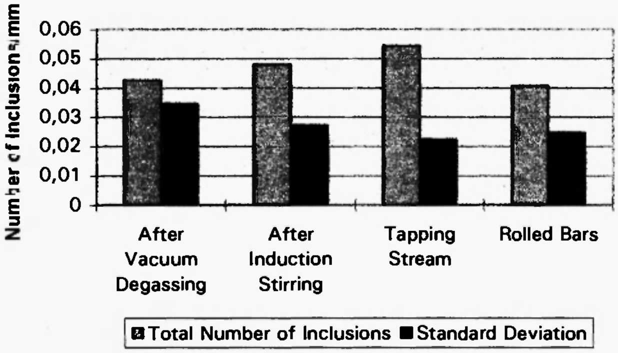

Fig. 5: Variation in DT-type inclusions from end of vacuum degassing to rolling.

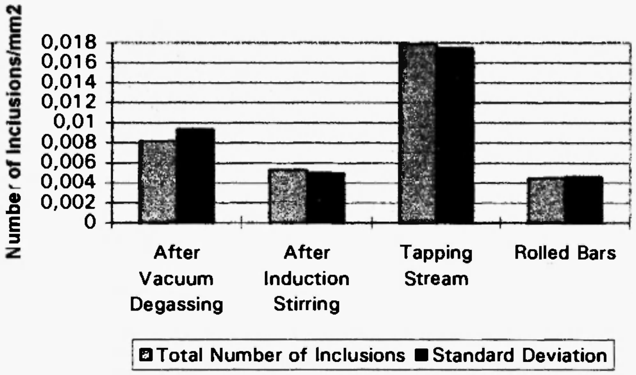

Fig. 6: Variation in DM-type inclusions from end of vacuum degassing to rolling.

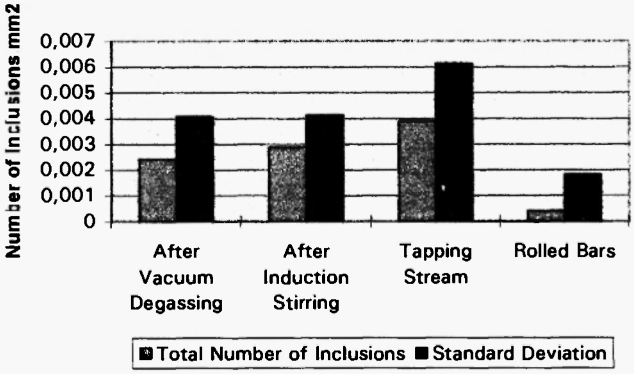

Fig. 7: Variation in DH-type inclusions from end of vacuum degassing to rolling.

number of light (DT) and of heavy (DH) inclusions. The average of the number of medium (DM) inclusions decreased after induction stirring, but increased in the tapping stream beyond the number of inclusions found before the induction stirring. The DM inclusion count again fell to below the number of inclusions found in the first sample once the steel had reached the rolled bar stage.

Inclusion Composition

The SEM analysis showed Al, Mg, Ca, Si, S, Mn, O, Fe, Cr, Ni, and Au to appear in different extents in different inclusions. The Fe, Cr, and Ni are part of grade steel composition, and were therefore disregarded. The Au, detected from the gold plating, was also disregarded. The remaining elements were then assumed to be oxides in the inclusions being scanned. Mn was not considered when evaluating the data, since it was averaged only 1.94 percent of the an inclusion's composition and was found in only 25 percent of the inclusions. Also, MnO is not known to have any ill effects on ball bearing steel properties. Table 1 displays the percentage of each inclusion type found, the average composition of each inclusion type, and the average size of the inclusions. The data is separated according to when in the steelmaking process the samples were taken. The samples taken after vacuum degassing contained on average 17.1% Al_2O_3-MgO inclusions, 39.0% Al_2O_3-MgO-CaO inclusions, and 43.9% Al_2O_3-MgO-CaO-S inclusions. The samples taken after induction stirring were on average 12.5% Al_2O_3-MgO, 45.0% Al_2O_3-MgO-CaO, 40.0% Al_2O_3-MgO-CaO-S, and 2.5% Al_2O_3-MgO-S. Finally, the samples from the tapping stream contained on average 9.7% Al_2O_3-SiO_2 inclusions, 9.7% Al2O3-MgO inclusions, 54.8% Al_2O_3-MgO-CaO inclusions, 16.1% Al_2O_3-MgO-CaO-S inclusions, 6.4% Al_2O_3-MgO-SiO_2 inclusions, and 3.2% Al_2O_3-MgO-CaO-SiO_2 inclusions. The average inclusion size was 3.4 µm. When present, the elements in the inclusions averaged about 74% for Al, 22% for Mg, and 2.5% for Ca. Notably, Si and S were never found in the same inclusion. A study by Wijk /11/ has shown that this is correct from a thermodynamic point of view, since SiO_2 in inclusions suppress the formation of CaS. Also, Si was only found in the samples taken in the tapping stream.

5. DISCUSSION

Total Oxygen Content

When analyzing the samples using optical microscopy it was found that both the tapping stream and the mold samples contained a large amount of pores. The corresponding analyses of the total oxygen content in these samples were also high. In the case of the samples taken in the tapping stream only 4 analyses were lower than 11 ppm and therefore the data from the tapping stream was excluded in the comparison.

The large effect of sampling method on the porosity of the sample and in turn on the analysis result suggests that tapping and ingot sample results can only be used for rough comparisons with the ladle samples when studying total oxygen content. The ladle samples can, however, be directly compared since the same sampling method was used after vacuum degassing and after induction stirring.

When comparing the ladle samples, it can be seen that the total oxygen content remains almost the same. Thermodynamics shows that the 20°C increase during induction stirring should cause a 1.1 ppm increase in the dissolved oxygen capacity of the steel. This would lead to the formation of new, primarily small inclusions (DT) if the sample was then cooled, as it is during sampling. However, the induction stirring promotes collisions of inclusions and separation of inclusions to the top slag, thus removing oxides from the steel. Hence overall, the total oxygen content seems to remain the same after the final induction stirring and heating treatment.

Inclusion Size Distribution

Steel making is not a precision process with respect to inclusion characteristics, as can be seen by the standard deviations in Figures 4, 5, 6, and 7. On top of deviations stemming from the production process, sampling procedures are also a source of discontinuities. Even if the samples in the ladle are taken using an automatic sampling system which always samples at the same location, the distribution of the inclusions in the ladle might be non-uniform.

During the final induction stirring and heating period, the total number of inclusions increases slightly. As mentioned earlier, the 20-degree heating of the steel should increase the dissolved oxygen content by 1.1 ppm. As the steel sample solidifies, this dissolved oxygen should react with aluminum, which has the strongest affinity to oxygen among the elements in the steel, to form small DT alumina inclusions. This reaction could partially explain the increase in DT-type inclusions in the samples taken after the final treatment period. The decrease in the larger DM-type inclusions during the same period could be explained by the effect of induction stirring, which promotes the collision of smaller inclusions to form larger inclusions as well as the rising of the larger inclusions towards the upper part of the ladle where they can become assimilated into the top slag. The decrease in DM inclusions does not, however, outweigh the increase in DT inclusions, and the total number of inclusions thus increases during the final ladle treatment. This increase suggests that the current of 1000 A which is presently used during induction stirring is too high. A high current allows reoxidation from the top slag and from the refractory to take place, transporting more oxygen into the steel where it reacts with aluminum to form more small inclusions. These results points towards trials being carried out using a lower current during induction stirring in order to minimize the reoxidation and thereby lower the total number of inclusions.

Most likely, a large amount of reoxidation is occurring during sampling in the tapping stream. This statement is supported by comments from the operators saying that if the sampler is held towards the outer edge of the stream, instead of in the middle, air can be sucked into the sample. From their experience, the total oxygen content in samples taken at the outer edge of the stream is higher than what is normal. Since the medium (DM) inclusion content was quite high, it seems likely that new DT inclusions collided and formed larger inclusions. The high pressure and turbulence from the sampling stream may have enhanced the colliding of the DT inclusions.

As the steel cools in the ingot, the solubility of the oxygen in steel decreases and new inclusions are formed. Despite this, many inclusions are believed to be separated to the upper part of the mold during solidification The number of inclusions from the samples taken in the mold in this investigation could not be analyzed because the sample quality was too poor. Therefore, it was not possible to determine the inclusion content in the mold during the initial stages of solidification. However, when comparing the total number of inclusions in the ladle samples with the samples from the rolled bars, it is clear that the number of inclusions has decreased during the casting operation. There are a couple of contributors to this decrease. First, an exothermic top and an insulating powder in the ingot mold help the steel extend the liquid stage. In this way, the steel cools quite uniformly. Also, natural convection due to cooling of the mold walls causes a downward flow along the walls and an upward flow in the middle of the ingot. The slow solidification gives inclusions more time to collide, and the upward flow in the middle of the ingot promote the separation of the inclusions to the top part of the ingot where they can be assimilated into the mold flux. Finally, the top part of the ingot is cut off and not used in making the rolled bars, thus removing the most inclusion rich section from the finished product.

Inclusion Composition

Three types of inclusion compounds exist in the steel after vacuum degassing: Al_2O_3-MgO-CaO with sulfur (43.9%), Al_2O_3-MgO-CaO without sulfur (39.0%), and Al_2O_3-MgO (17.1%), Table 1. According to the phase diagram of the Al_2O_3-MgO-CaO system in Figure 8, three oxides can exist in the solid solution spinel (Ca,Mg)O-Al_2O_3. The composition of the spinel can change along the line MA (MgO- Al_2O_3) to CA (CaO-Al_2O_3), depending on the activity of CaO. 17.1% of the inclusions were of the MA-type solid solution spinels and 43.9% of the MA-type with a small amount of CaO dissolved. An indication of that these inclusions actually exists as solid solutions is given in Figure 9. A SEM mapping picture of a 5.5 micrometer inclusion shows that the aluminum and magnesium are homogeneously distributed in the inclusion. Calcium is also almost homogeneously distributed, but since the CaO content is also only 1.7% it is difficult to make an exact assessment. From the SEM picture in Figure 9 it is also obvious that the magnesium area is smaller than the sulphur and calcium area. This is a clear evidence that the three-component solid soution spinel is surrounded by a CaS layer.

The inclusion types and compositions changes very little during the final induction stirring and heating period (Table 1). However, a new type of inclusion occurs which consist of 77.3% Al_2O_3, 22.7% MgO and sulphur, but it is not as frequent (2.5%). For this type of inclusions the spinel is surrounded by a MgS layer,

Table 1

Variation in inclusion compositions from after vacuum degassing to the tapping stream. The standard deviation of the analyses are given within the parentheses.

After vacuum degassing:

	% Al_2O_3	%MgO	%CaO	size (μm)	% of tot incl's
Al_2O_3-MgO	77,31 (+-0,89)	22,69 (+-0,83)		3,32 (+-0,89)	17,1
Al_2O_3-MgO-CaO	75,82 (+-1,29)	22,31 (+-0,42)	1,70 (+-0,89)	3,42 (+-1,40)	39,0
Al_2O_3-MgO-CaO-S	68,76 (+-9,39)	20,75 (+-3,80)	5,06 (+-6,64)	3,37 (+-1,09)	43,9

After induction stirring:

	% Al_2O_3	%MgO	%CaO	size (μm)	% of tot incl's
Al_2O_3-MgO	77,22 (+-0,66)	22,78 (+-0,66)		2,87 (+-0,51)	12,5
Al_2O_3-MgO-CaO	76,34 (+-0,62)	22,09 (+-0,79)	1,41 (+-0,52)	3,52 (+-0,91)	45,0
Al_2O_3-MgO-CaO-S	72,76 (+-4,99)	20,90 (+-1,46)	3,32 (+-2,03)	3,32 (+-0,74)	40,0
Al_2O_3-MgO-S	77,27	22,73		2,6	2,5

Tapping stream:

	% Al_2O_3	%MgO	%CaO	%SiO_2	size (μm)	% of tot incl's
Al_2O_3 -SiO_2	63,18 (+-18,20)			36,85 (+-18,22)	4,55 (+-2,05)	9,68
Al_2O_3-MgO	77,75 (+-1,03)	22,25 (+-1,03)			4,13 (+-1,94)	9,68
Al_2O_3-MgO-CaO	76,99 (+-1,98)	21,90 (+-0,87)	1,70 (+-0,72)		3,2 (+-1,06)	54,84
Al_2O_3-MgO-CaO-S	74,58 (+-2,84)	21,97 (+-0,74)	2,39 (+-1,11)		3,58 (+-1,21)	16,13
Al_2O_3-MgO- SiO_2	66,18 (+-2,90)	20,28 (+-5,36)		13,54 (+-8,25)	5,0 (+-0,00)	6,45
Al_2O_3-MgO-CaO-SiO_2	75,76	21,58	1,96	0,7	2,5	3,23

instead of a CaS layer as shown earlier in Figure 9.

Also notable is that the standard deviation of the analyses is quite low, especially for the inclusion compounds that do not contain sulfur (0.42% to 1.29%). The conclusion from the result of the low standard deviation is that the secondary refining process that is carried out after vacuum degassing must be quite stable. It is possible from one production heat to the next, to produce a steel with almost the same inclusion composition. The higher standard deviations in the cases of the inclusions containing sulfur may be explained by sulfur always being present as a CaS or MgS layer around the inclusion. Since sulfur is not homogeneously distributed in the inclusion, the SEM analyses might give a faulty answer. It is hard enough to analyze inclusions as small as 2 to 4 microns using a SEM with an EDS attached to it, since it is difficult to directly hit the inclusion during the composition determination.

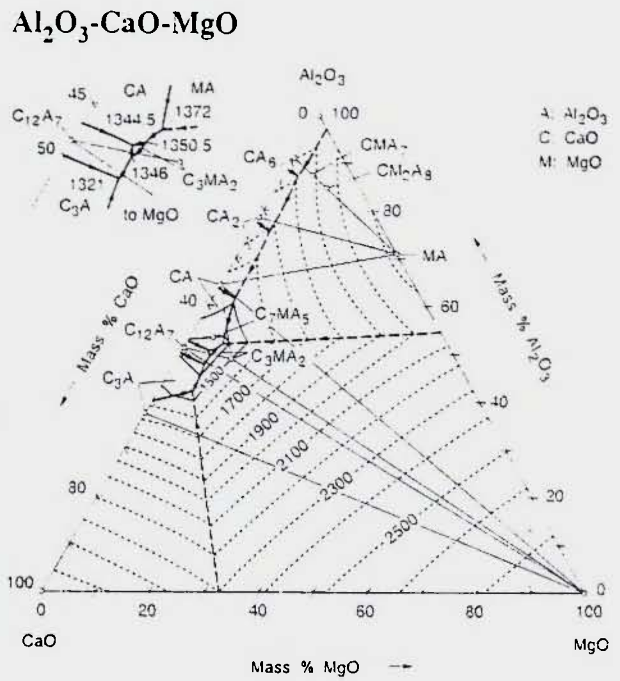

Fig. 8: Phase diagram of the Al_2O_3-MgO-CaO system /12/.

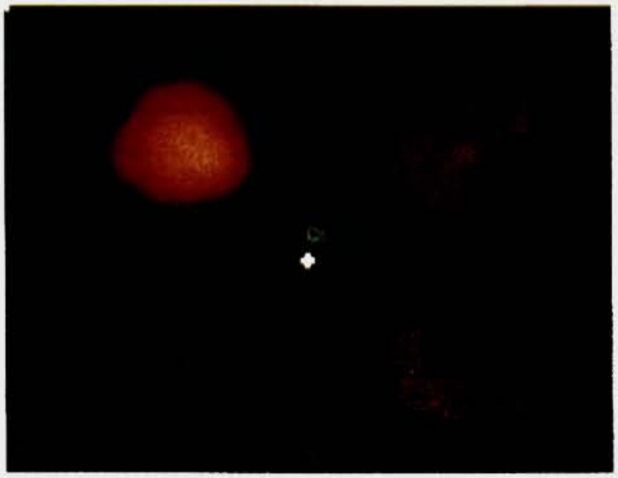

Fig. 9: SEM mapping picture of a 5.5 mm Al_2O_3-MgO-CaO inclusion surrounded by a CaS layer.

The samples from the tapping stream contain a larger variety of inclusion types. Besides the inclusions found in the ladle, Al_2O_3-SiO_2 and Al_2O_3-MgO-SiO_2 inclusions are also common. The silica oxides are probably formed when the steel is reoxidized by air. As mentioned earlier, it seems as if quite a lot of air was entrapped into the steel during the tapping stream sampling. This high amount of oxygen from the air allowed not only aluminum, but also the silicon in the steel to react with the dissolved oxygen to form new oxides. So many oxides formed and the turbulence was so high, that the inclusions collided and, for example, compounds consisting of both alumina and silica were formed. The average size of the analyzed tapping stream inclusions was also larger than the average sizes found in the ladle samples. These results agree with the earlier findings that the number of DM inclusions increases in the tapping stream (Figure 6).

6. CONCLUSIONS

The specific conclusions from this study may be summarized as follows:

1. Samples taken in the tapping stream using RS samplers are not suitable for determination of inclusion characteristics since the reoxidation from air during sampling gives rise to an unrealistically high inclusion content and inclusions containing silica which were previously not found.
2. Samples taken in the mold using vacuum tube samplers are so porous so it is not possible to evaluate the inclusion characteristics.
3. There is not a large change in the total oxygen content and the total number of oxidic inclusions in the ladle during the final induction stirring and heating period. Hence, the effect of the stirring on decreasing the oxygen content and the number of oxidic inclusions fails to outweigh the effect of heating the steel.
4. Three main types of inclusions were found in the ladle after vacuum degassing: Al_2O_3-MgO-CaO with sulphur (43.9%) and without sulphur (39.0%), and Al_2O_3-MgO (17.1%).
5. The secondary refining process in the ladle must be quite stable with respect to inclusion composition, since the standard deviations of analysed inclusion compositions were relatively low (less then 1.29% for inclusions containing no sulphur).

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