The Effect of Ferromanganese Cleanness on Inclusions in Steel

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1 ABSTRACT

The effect of ferromanganese additions on non-metallic inclusions in steel have been studied in laboratory and full-scale experiments. Non-metallic inclusions present in ferromanganese and steel samples were characterised by optical microscopy, scanning electron microscopy (SEM) and microprobe technique. Number per area unit and size distributions of non-metallic inclusions were assessed according to the SS 111116 method. Characteristics of non-metallic inclusions have been determined for six different ferromanganese qualities. The most common compositions were MnO or complex MnO-MnS-SiO_2. The number per area unit was found to decrease with an increased carbon content in the ferromanganese. Laboratory trials were carried out using the same six qualities to determine the effect on steel cleanness. Again, the number of non-metallic inclusions in the steel was found to decrease with an increased carbon content in the ferromanganese. The composition of these were mainly MnO and MnS. Finally, full-scale experiments were carried out with four of these ferromanganese grades. Three minutes after addition of ferromanganese the number of inclusions in the steel samples showed no obvious relation to the cleanness of the ferromanganese used. However, the analysis of the inclusions show high levels of MnO and MnS present in the non-metallic inclusions.

2 INTRODUCTION

Non-metallic inclusions present in steel can be both beneficial and/or detrimental to material properties of the finished product. An example of a beneficial inclusion type is manganese sulphide. Its presence in machining steels facilitates increased machining rates and may also extend tool life.[1] On the other hand, presence of detrimental inclusions like titanium carbon-nitrides may override any improvement in physical and mechanical properties obtained by having an optimal microstructure.[2] Since solid steel have a limited solubility of oxygen, nitrogen and sulphur, both detrimental and beneficial inclusions will always be present in commercial steel products. However, for applications with high demands on material properties, clean steels with low levels of non-metallic inclusions are an absolute requirement.

To achieve these requirements, it is important to study the mechanisms of inclusion formation and removal during steelmaking. Recently, the research on non-metallic inclusions in steel has mainly been concentrated on slag-metal reactions, deoxidation practise, inclusion modification and inclusion removal.[1,3-5] Overall, only a limited number of investigations concerning the effect of ferroalloy additions on inclusion characteristics in steel have been published. For example, Wijk and Brabie[6] studied the effect of ferrosilicon, while Schöberl and Straube[7] studied effect of ferrochromium. In another study, Molins et al.[8] evaluated the effect of impurity elements present in different ferroalloys. Reviews in this area also indicates a need for further research.[9,10]

The present investigation aims to extend the knowledge about inclusions present in commercial low (max. 0.5 wt-% C) and medium carbon (max. 1.5 wt-% C) ferromanganese and the influence on steel cleanness. The investigation has been divided into three distinct

parts. First, inclusions and impurity elements present in ferromanganese were studied. Second, laboratory experiments were performed to evaluate possible influence of ferromanganese additions on the inclusion characteristics in steel. Finally, to verify these results, full-scale experiments were carried out using four of the originally six ferromanganese grades. Parameters that were studied in these investigations includes quantity, size distribution, shape and composition of non-metallic inclusions in ferromanganese and steel samples. Efforts were also made to determine if there is a relationship between the inclusion characteristics in the steel and the ferromanganese grade used.

3 METHODS AND PREPARATIONS

3.1 SAMPLING

For the microscopic examination and laboratory experiments, as-cast ferromanganese was gathered at the production sites. Liquid steel samples were taken with 5.8 mm quartz tubes during the laboratory experiments. Immersion samples with 6 mm thickness and LSHR (Liquid Sampling - Hot Rolling) samples were taken using an automatic sampler during the plant trials.

3.2 SAMPLE PREPARATION

Before microscopic examination the samples were encapsulated in a conducting phenolic mounting compound (Buehler® Konductomet® I) with a visible surface area of at least 200 mm². The mounted samples were then polished with silicon carbide papers or a diamond-polishing disc. Finally, a textile disc with diamond spray (3μm) was used to obtain a scratch free surface. After completed polishing, the samples were stored in a dessicator with a moisture absorbing agent.

3.3 ANALYTICAL METHODS

Evaluation of the samples was performed by chemical analysis, micro-inclusion size assessment using the SS111116 method, and micro-inclusion composition assessment by SEM and microprobe. Moreover, to determine the macro-inclusion content the LSHR samples were examined by ultra-sonic testing. In the following, these methods will be described more in detail.

3.4 CHEMICAL ANALYSIS

The samples were analysed for oxygen and nitrogen content by combustion (Rosemont NOA 5003) with a relative accuracy of ± 1%. Carbon and sulphur were analysed by combustion (Rosemont CSA 5003) with a relative accuracy of ± 1% and ± 3%, respectively. X-ray fluorescence analysis was used to determine the contents of Mn, Fe, P and Si.

The results of the chemical analysis of used ferromanganese is presented in **Table I.**[11] Note that the grade numbers A to F will be used throughout this paper, and that the same ferromanganese samples were used for both the metallographic study of ferromanganese as well as the laboratory scale experiments.

No.	Grade	Mn	Fe	C	S	O	N	P	Si
A	LC 0,5% C	79.6	17.7	0.34	0.004	1.11	0.14	0.17	0.01
B	MC 1,0% C	81.1	16.5	0.77	0.005	0.89	0.06	0.17	0.12
C	MC 1,5% C	81.5	15.9	1.52	0.003	0.01	0.07	0.16	0.04
D	MC 1,5% C	81.0	16.1	1.38	0.003	0.02	0.07	0.16	0.39
E	LC 0.5% C	81.4	17.3	0.48	0.003	0.04	0.04	0.16	0.32
F	MC 1.5% C	80.6	16.8	1.41	0.003	0.12	0.06	0.16	0.29

Table I - Composition in weight-% of used LC and MC ferromanganese. Grades A to C are standard refined ferromanganese and grades D to F are low oxygen ferromanganese.[11]

3.5 MICRO-INCLUSION ASSESSMENT

Assessment of the inclusions in all samples has been performed according to the SS 111116 method.[12] This method was developed to ensure that all operators counting inclusions from the same sample would obtain the same results.[13,14] When using this method, all inclusions are characterised by their geometry and size according to a picture chart presented in the Swedish standard SS 111116.[12] Four size categories are used: T comprises apparent particle diameters between 2.8 and 5.6 μm, M diameters between 5.6 and 11.2 μm and H diameters between 11.2 and 22.4 μm. For inclusions larger than 22.4 μm in diameter, their size is noted individually

A computer software, PC MIC©, was used to assess the collected data. This program allows the user to set the visual field diameter, number of scale divisions within the visual field diameter and magnification. After the inclusions in a given sample have been assessed, the program calculates the characteristic parameters for the different geometrical classes of inclusions according to the algorithms presented in SS 111116.[12] Parameters used in this study are the number and area percentage per square millimeter of inclusions.

For all assessments in optical microscope, a magnification of 200x was used combined with a visual field of 0.8 mm. A total area of at least 160 mm² was examined on each of the samples.

3.6 SCANNING ELECTRON MICROSCOPE

Two different scanning electron microscopes have been used during the course of this investigation to determine the composition of non-inclusions. The steel samples taken during the laboratory experiments were examined using a JEOL JSM6300 with EDS, while the samples from the plant trials were studied using a JEOL JSM840 with EDS.

During analysis, acceleration voltage was set to 20 kV with a working distance of 25 mm. The live time for each analysis was set to at least 75 seconds. Calibration against a cobalt reference was performed at two hours interval.

These analyses includes both qualitative analysis (element mapping) and quantitative microarea analysis. The quantiative analyses were subsequently re-calculated by using the ZAF correction method with three iterations.

3.7 MICROPROBE

Some of the samples have been analysed using a JEOL JXA-8900M WD/ED combined microprobe to determine the constituent elements of the different inclusions. This equipment has the advantage of being able to quantify elements with low atomic weights, e.g. carbon, nitrogen and oxygen. The analysis of the inclusions included both qualitative analysis (element mapping) and quantitative microarea analysis.

3.8 MACRO INCLUSION ASSESSMENT

All LSHR samples were first hot rolled to a height of about 4 mm, and then straightened and normalised. The middle part of the plate, was removed and milled down to a rectangular plate with level surfaces and a final measurement of 300x4x25 mm. Using a Krautkramer Branson USIP20 HR with a high frequency equipment these specimens were subject to ultrasonic high frequency testing submerged in a water-filled tank. By using flat bottom hole specimens, the equipment was calibrated at three ranges of defect sizes. The ranges of defect sizes that were detected are 35 to 59 μm, 60 to 85 μm and larger than 85 μm. Further information regarding the LSHR method can be obtained from references 15 to 17.

4 EXPERIMENTS

4.1 LABORATORY EXPERIMENTS

In principle three different commercial grades of refined ferromanganese, namely LC FeMn with max. 0.5 wt-% C, and two grades of MC FeMn

with max. 1.0 wt-% and 1.5 wt-% C, respectively, were studied. In accordance with **Table I**, grades A to C are standard refined ferromanganese while grades D to F are low oxygen ferromanganese. Two alloying experiments were carried out for each of the six ferromanganese grades. The experimental setup is pictured in **Figure 1**.[18]

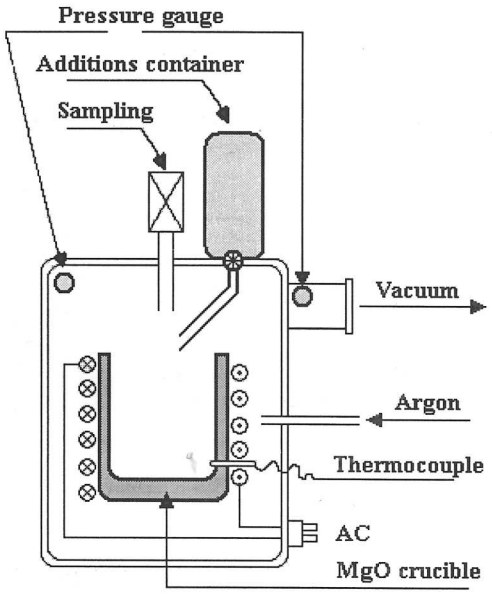

Figure 1. Experimental setup of laboratory scale experiments.[18]

The experiments were carried out in a Balzer VSG50 vacuum induction furnace. A magnesite crucible was used to hold the melt at a temperature of 1600°C. This was measured by a type B thermocouple which was connected to a PID regulator to control the temperature within ± 1°C. For each experiment, 20 kg of pure iron was placed in the crucible and thereafter the vacuum chamber was sealed and evacuated to about 0.09 torr (~ $1.2 \cdot 10^{-4}$ atm). Thereafter the chamber was purged with pure argon (< 5 ppm O2 and < 5 ppm H2O) and induction heating was started. At a temperature of about 400°C, the vacuum chamber was again evacuated to further decrease the oxygen content in the atmosphere. Then the chamber was filled with pure argon to obtain atmospheric pressure inside the vessel. Thereafter, argon was introduced at a rate of 30 Nl/min to the vacuum chamber to keep the atmosphere inert during the experiment. Additions of pure carbon, pure aluminium and ferromanganese were made under inert atmosphere through an evacuated and purged additions container. Temperature and pressure were monitored during the whole experiment.

The additions that were made prior to the ferromanganese addition included pure carbon and pure aluminium. At the start of the experiment, 37.5 ± 0.5 g pure carbon (> 99.97 wt-% C) was added to reach a carbon content of about 0.18 wt-% C. Nine minutes later, an addition of 47.4 ± 0.1 g aluminium (> 99.6 wt-% Al) was made, corresponding to 0.14 wt-% Al. At 15 minutes from the start of the experiment, ferromanganese corresponding to 1.45 to 1.60 wt-% Mn was added. During the experiment, four liquid steel samples were taken by suction with quartz tubes with an inner diameter of 5.8 mm. These samples were taken at six-minute intervals, i.e. at 6, 12, 18 and 24 minutes. Each sample rod was then cut into small cylinders in preparation for chemical analysis and metallographic examination.

4.2 FULL-SCALE EXPERIMENTS

Four ferromanganese grades (A,B, E and F) were tested in full-scale at Ovako Steels production site in Hofors, Sweden. These grades were chosen following the metallographic and laboratory studies. Five experiments were performed for each of the four grades. During the experiments, all primary additions were made before the addition of ferromanganese, e.g. deoxidants, other ferroalloys, synthetic slag components. A reference sample was then taken before the addition of ferromanganese. This was considered necessary to ensure that any major differences in content and composition of inclusions between the different experiments could be determined. About three minutes after ferromanganese was added, a second sample was taken followed by two more samples taken at three minute intervals. A fourth and fifth sample was taken directly after vacuum degassing and before casting. Immersion samples with 6 mm thickness were taken

according to this procedure for every experimental heat. In addition, during one experiment for each of the ferromanganese grades, LSHR-samples were taken at the same time. All samples were taken by using an automatic sampling equipment with a given sampling time and sampling depth into the molten steel.

5 RESULTS AND DISCUSSION

5.1 INCLUSIONS IN FEMN

In the metallographic investigation on the ferromanganese grades presented in **Table I**, it was found that the most common inclusion composition is manganese oxide, followed by complex inclusions also containing manganese sulphide and/or silicon oxide. These inclusions were, in average, smaller than 6.7 µm. Also, the pure manganese oxides were found present in two different shapes, dendritic and rhombic. In an attempt to explain the difference between these two types, several analyses were made. The results of these are presented in **Table II**. It shows that the rhombic type of manganese oxide has a substantially higher content of silicon and titanium. **Figure 2** and **3** shows microprobe element mappings of these two inclusion types.

Element	Mn	Al	Si	O	Ti	Remark
Average	48.5	0.02	0.01	51.3	0.0	Dendritic
Std. Dev.	0.2	0.01	0.01	0.2	0.0	Dendritic
Average	47.8	0.06	0.34	51.3	0.19	Rhombic
Std. Dev.	1.0	0.05	0.31	0.7	0.07	Rhombic

Table II - Element analyses in at-% of inclusions present in LC and MC ferromanganese.[11]

Results from the inclusion assessment are shown in **Figure 4**. It is evident that standard refined low-carbon ferromanganese has the highest inclusion content of all grades studied.

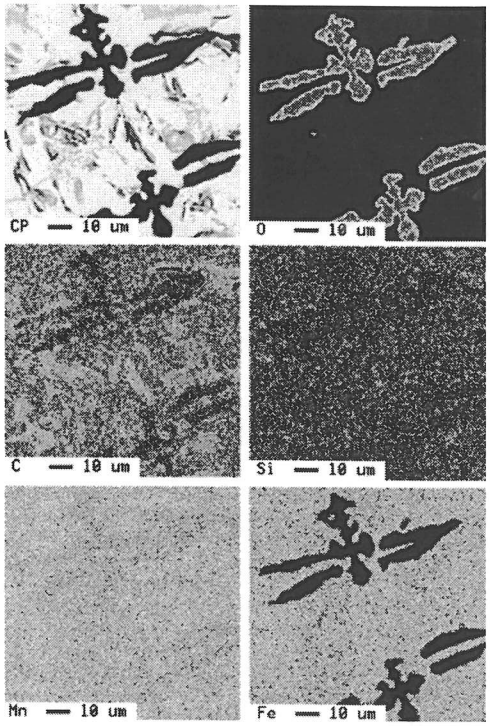

Figure 2 - Dendrite shaped MnO found in grade B ferromanganese. Average composition is 48.5% Mn and 51.2% O.[11]

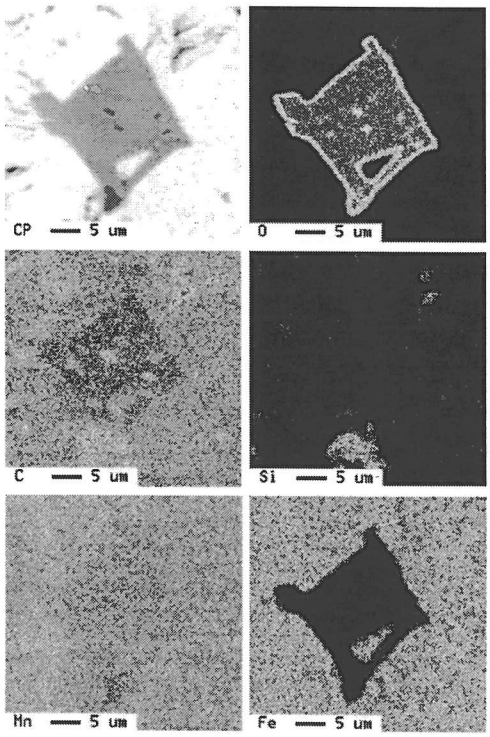

Figure 3 - Rhombic shaped MnO found in grade C ferromanganese. Average composition is 47.8 at-% Mn and 51.3 at-% O.[11]

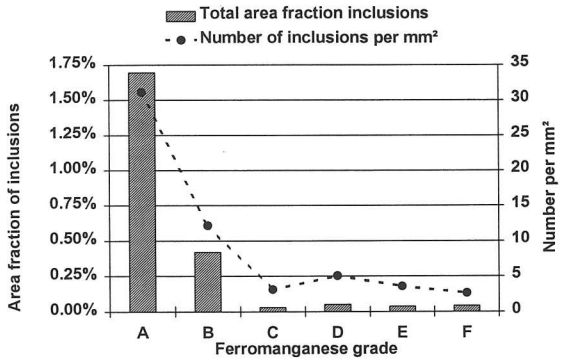

Figure 4 - Area fraction and number of inclusions per mm² in grades A to F.[18]

5.2 LABORATORY EXPERIMENTS

The results of the micro-inclusion size assessment of final steel samples are presented in **Figure 5**. For the steel samples from experiments with grades A, B and C, the total area fraction of inclusions follow an inverse relationship with the carbon content of the ferromanganese. This relationship is, however, not true for the three low oxygen ferromanganese grades, D, E and F.

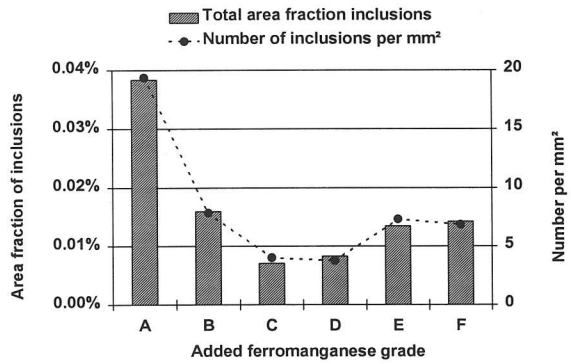

Figure 5 - Area fraction and number of inclusions per mm² in final steel samples.[18]

A comparison of the inclusion contents in ferromanganese and steel samples, show that there are similarities between the results for all grades. A high inclusions content in the added ferromanganese has resulted in a higher content in the final steel sample. However, the difference between the highest and lowest area fraction of inclusions is lower for the steel samples than for the ferromanganese samples.

There is also a notable difference between the two low carbon grades with max. 0.5 wt-% C, grade A and E. In this case, low oxygen ferromanganese with max. 0.5 wt-% C result in a considerably lower amount of inclusions in the steel (0.014 area-%) compared to corresponding experiments using standard refined ferromanganese with max. 0.5 wt-% C (0.038 area-%).

Micro-inclusion composition assessment was made for the final steel samples. At least 15 inclusions from each of the samples were quantitatively analysed in SEM. **Table III** presents an overview of the analysed inclusions. From these results it is evident that the inclusions mainly consist of MnO and MnS.

It is of interest to compare the composition of inclusions found in the steel to see if they have some resemblance with those found in ferromanganese. In the literature, inclusions in ferromanganese have been reported to mainly consist of MnO, MnS and SiO_2.[11,19-22] Over 85% of the total number of inclusions have been found to consist of pure MnO.[11] It is evident from **Table III** that the inclusions in the steel samples have a similar composition to those in ferromanganese. An exception is the alumina containing inclusions, which are present in steel but extremely scarce in ferromanganese. This is expected since the steel was deoxidised with aluminium.

Composition group	Added ferromanganese grade.					
	A	B	C	D	E	F
A	6	5	2		4	6
B	9	9	7	11	11	6
C	3	1			1	1
D		1	1	1	1	

Composition groups:
A: 80-100% MnO+0-20%MnS
B: 80-95%MnS+20-5%MnO
C: 70-100%Al_2O_3+0-15%MnO+0-10%SiO_2
D: 0-10%Al_2O_3+70-80%MnO+10-30%MnS

Table III - Number of inclusions in each composition group in final steel samples from laboratory scale experiments.[18]

Knowing that inclusions found in ferromanganese are similar in composition to those found in the steel, it is of interest to ascertain if the inclusions found in steel originate from ferromanganese or if they are dissolved and later formed in the steel. As mentioned above, over 85% of the inclusions found in ferromanganese consist of pure MnO.[11] Since the melting point of pure MnO is 1844°C[23] and the steel temperature during the experiments was fixed at 1600°C, there is a good chance that the MnO inclusions from the ferromanganese will remain in the steel after the addition. However, the larger inclusions will probably rapidly be separated the top of the melt and to the refractory. It should also be noted that if the MnO inclusions are not pure and, for example, contain MnS, the melting point will decrease rapidly.

5.3 Full-Scale Experiments

Micro-inclusion size assessment with SS111116 were made on samples from three heats for each of the studied ferromanganese grades. The grades studied in full-scale were A, B, E and F in accordance with Table I. The manganese content in the samples were analysed to ensure that all ferromanganese had been dissolved before the sampling was made. The results showed that the ferromanganese alloy was dissolved at the time of the sampling. In other words, it can be assumed that the determined inclusion contents adequately represents the effect of the ferromanganese addition.

Figure 6 shows the average micro-inclusion content in the examined steel samples. For each of the ferromanganese grades, the average of three heats is indicated. Results marked (ref) are from the reference samples, which were taken before adding ferromanganese, and the results marked (1) are from the first steel sample after addition. The inclusion content is fairly constant for the reference samples. The average inclusion density the reference samples was 0.133 per mm^2 with a standard deviation of 0.021. A comparison between the experiments shows that the inclusion content increases for grades A, B and E. However, it is decreasing when using grade F.

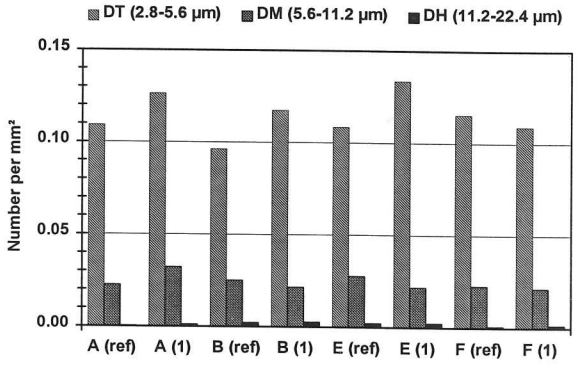

Figure 6 - Average number of inclusions per mm^2 in examined steel samples.[18]

Inclusions present in steel samples taken 3 minutes after the addition of ferromanganese have been analysed in SEM. About 10 inclusions from each of the samples were analysed and the results are presented in **Table IV**. The analysed inclusions were in the range between 2 and 10 µm in diameter.

	FeMn grade			
Type of inclusion	**A**	**B**	**E**	**F**
Al_2O_3	2	1		1
Al_2O_3-MnO	4	1	1	4
Al_2O_3-MnO-MnS	3	2	7	3
Al_2O_3-CaO-MnO	1	1	1	1
Al_2O_3-CaO-MnO-MnS	1	2		2

Table IV - Type of inclusions present in samples from full-scale experiments. These steel samples were taken after adding ferromanganese.

The presence of aluminium oxide (Al_2O_3) in the analysed inclusions, suggest that these originate from the aluminium deoxidation. However, since the steel had been deoxidised before the addition of manganese, the manganese oxide (MnO) present in some of the inclusions indicate that these inclusions may instead have been inherited from the ferromanganese.

In order to determine the content of macro-inclusions, the LSHR samples were subjected to

ultrasonic scanning submerged in a water tank. This technique allows for detection of inclusions larger than 35 µm. The results are presented in **Figure 7**, wherein each set of bars represent the results from one heat. Illustrated are the results from the reference sample (ref), taken before adding ferromanganese, and the first (1) and third (3) sample after addition.

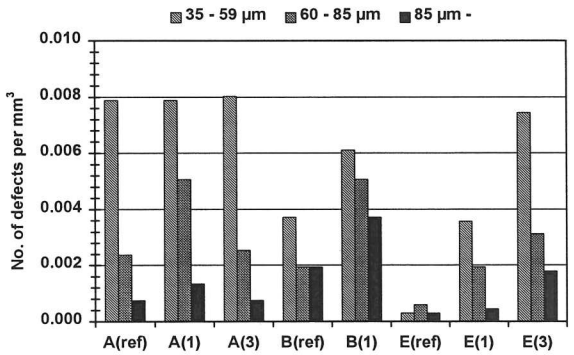

Figure 7 - Results from ultrasonic scanning of LSHR samples.

The results show that the number of macro-inclusions change during the course of the ladle treatment. For ferromanganese grade A, the number of inclusions per mm^2 between 35 and 59 µm remains unchanged throughout the treatment, while the number of larger sized inclusions changes during the treatment. But, there is virtually no difference in inclusion density before and after the addition of ferromanganese.

For grade B and E, **Figure 7** shows that the macro-inclusion density increases during the treatment. The cause for this is probably topslag entrainments during the heating stage of the ladle treatment. Thus, this result has no direct connection to the ferromanganese addition.

6 CONCLUSIONS

The influence of cleanness of six ferromanganese grades on inclusion characteristics in steel has been studied in laboratory and full-scale experiments. These experiments were preceded by a metallographic study of the added ferromanganese. The investigation was focused on the behaviour of micro- and macro-inclusions in ferromanganese and steel. The more specific conclusions from this study are:

- Results from micro-inclusion assessment of ferrromanganese show that dendritic MnO are in majority among inclusions in ferromanganese, followed by rhombic MnO and complex inclusions. Furthermore, it is concluded that for normal refined ferromanganese, grades A to C, the inclusion content is related to the carbon content of the sample. That is, a higher carbon content results in a lower inclusion content. Also, the difference in inclusion content between the low-oxygen grades (D to F) is very low even though the carbon content varies between 0.48 to 1.41 wt-%.

- The micro-inclusion population in commercial ferromanganese consists of single phase MnO and complex inclusions containing MnO-MnS-SiO_2. Manganese oxide inclusions are present as two different types, namely dendritic and rhombic. Of these it is concluded that the rhombic type contains more impurity elements like Al, Si and Ti. This implies that rhombic manganese oxides are presumably formed in the residual melt which has a higher degree of contamination.

- Results from the laboratory experiments showed that the fraction of inclusions in the steel follow an inverse relationship with the carbon content of the ferromanganese for standard refined grades, grades A to C. Low oxygen ferromanganese with max. 0.5 wt-% C result in a considerably lower amount of inclusions in the steel compared to corresponding experiments using standard refined ferromanganese with max. 0.5 wt-% C. It is therefore concluded that if ferromanganese has a high inclusion content, this will result in a higher inclusion content in the steel.

- A majority of the inclusions found in steel contain MnO and/or MnS, similar to the compositon of the micro-inclusions in ferromanganese. Note that the inclusion population in steel contains more MnS as well as alumina inclusions than ferromanganese. It can be concluded that pure MnO inclusions in the ferromanganese might remain in the steel after alloying.

- Full-scale experiments showed that the content of micro-inclusions between 5.6 and 22.4 μm remained unchanged, while the content of inclusions between 2.8 to 5.6 μm slightly increased for grades A, B and E. In contrast, the content decreased in the experiments with grade F. Furthermore, the macro-inclusion assessment shows no conclusive evidence for an effect of ferromanganese addition on the macro-inclusion population.

- The micro-inclusions composition in the steel samples from the full-scale experiment is very similar to the findings in laboratory scale. All analysed inclusions contained Al_2O_3 and, in addition, also MnO and in some cases MnS and CaO. The high content of MnO in the majority of the inclusions indicate the possibility that these have been inherited from the ferromanganese.

The results from the full-scale experiments indicates that ferromanganese has a small influence on inclusion characteristics in steel. It is possible that this influence may be important to certain steelmakers. Therefore, further investigations are needed in order to gain a better foundation from which more specific conclusions can be made.

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