

DISSOLVED IMPURITIES AND INCLUSIONS IN FESI AND SI, DEVELOPMENT OF A FILTER SAMPLER

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ABSTRACT

A literature review is presented on the solubility of the elements X ($X = O, N, C, H, Ca, Al$) in Si and FeSi. Solubility data from this study together with thermodynamic data are used to calculate the Gibbs energy change, ΔG_x° , for the solution of these elements to 1 mass% in liquid silicon. The interaction coefficients e_x^{Fe} have been derived for C, H and O.

A filter sampler of graphite for inclusions has been developed. With this sampler it is possible to describe the inclusion number size distribution; the number density [m^{-3}] versus the diameter of the inclusion [μm], in high temperature (1400 - 1700 °C) carbon-saturated melts such as Si and FeSi. Metal is drawn through the filter employing a vacuum pump. The concentration and size distribution of inclusions were obtained using image analyses. Composition of the inclusions in the filter samples was analysed by electron microscopy to be SiC and oxides of Al, Ca and Si.

INTRODUCTION

Critical for the development of new and improved steel grades is a low and controlled content of unwanted dissolved elements and inclusions. An example of dissolved elements greatly affecting the final steel quality is S, P, O, N, C, H in deep drawing steels [1]. Another example is steel sheets for electro-engines where nitrides and carbides have a detrimental effect [2]. With stricter requirements on "clean steel", impurities from alloy additions will become more and more unacceptable. Dissolved elements and non-metallic inclusions in the ferroalloy can increase the total level of unwanted elements and inclusions in steel and be detrimental to the final mechanical properties. Reported problems with nozzle blockage when employing special casting methods for ferroalloys may be due to inclusions. Obviously it is important to expand our knowledge concerning impurities in ferroalloys such as FeSi.

A literature review is presented on the solubility of the element X ($X = O, N, C, H, Al$) in Si and FeSi. Solubility data from this literature survey together with thermodynamic data from [16] are used to calculate Gibbs energy, ΔG_x° , for the solution of the elements to 1 mass% in liquid silicon for the above elements.

Metallographic investigation of cast samples can be excessively time and labour consuming, especially when the concentration of inclusions is low. A filter sampler allows direct sampling of relatively large volumes of liquid metal. This method has been used in the aluminium industry since the late seventies [3], but - has to the authors knowledge - never been adapted to ferroalloys. For inclusion investigation in ferrosilicon a vacuum-filtration sampler of graphite has been developed for high temperature (1400 - 1700 °C) melts. Employing a vacuum pump, up to 1.5 kg of ferrosilicon is drawn through the filter. The filter samples are sliced and polished before metallographic examination. The concentration and size distribution of inclusions are then determined using image analyses [4].

REVIEW OF SOLUBILITIES IN FESI AND SI

1. Solid solubility

The level of trace elements in commercial ferrosilicon depends on the choice of raw materials and on the production route (ladle treatment and casting method). Table I gives typical values for some trace elements in regular FeSi75 (FeSi75 = Ferrosilicon with 75 mass% Si). The levels of Al and Ca depend on the raw material source and if any, on how much slag is formed (production of FeSi and Si is, in principle, a slag free process) [5]. These two elements can be lowered by employing liquid metal refining.

Ti is present in all of the raw materials used and enters quantitatively into the metal, whereas the elements from groups V -VIIIb mainly come from the iron source. These elements are difficult to remove by refining techniques.

As a rule of thumb, there is almost no solid solubility in silicon. Two exceptions are B and P. The solid solubility of B in Si at $t_{\text{eut.}} = 1385^\circ\text{C}$ is 3 at% (~1.2 mass%) [6]. For P the corresponding value is 2.4 at% (~2.6 mass%) at 1130°C [7]. Solid solubility of Mg and Ca is reported to be 0.024 mass% and 0.016 mass% at 1200 and 1250°C respectively [8]. All these elements are completely soluble in the liquid state. During solidification of the ferroalloy the dissolved elements may form various inclusions and intermetallics. It is realized that it is important to know the solubilities when studying the presence of inclusions.

TABLE I Typical level of trace elements in FeSi75 (mass%)

Al	0.9
P	0.015
Ca	0.15
Ti	0.08
V	0.006
Cr	0.009
Mn	0.035
Ni	0.007
B	0.002
O	0.1
H	0.0005
N	0.0015
S	0.005
C	0.04

2. Liquid solubility of O, N, C and H in Si.

In the temperature range up to 1700°C these elements are all soluble to a small extent in liquid silicon and silicon-rich iron-silicon alloys and the solutions can therefore be considered as diluted up to the saturation limits. Except for hydrogen, which does not form any stable compound with silicon at these temperatures, these limits are defined by the formation of solid compounds of known thermodynamic properties, such as SiO_2 (crist.), Si_3N_4 and SiC . Thus, if the solubility of component X as defined by an equilibrium of the following type



is known, the standard Gibbs energy change for the solution of X in pure liquid silicon for the reaction



can be obtained as follows

$$\Delta G_{\text{X/X}}^0 = \frac{1}{m} \Delta G_{\text{Si}_n\text{X}_m}^0 - 2.303 RT \log[\%X] \quad (3)$$

This equation has been solved by the various investigators who have studied these systems by use of relative standard Gibbs energy data for the formation of Si_nX_m as obtained from various sources. In order to make the results more consistent and compatible with the database applied in computer calculations [16], we will, by use of the most reliable solubility data, recalculate these properties in the following.

2.1 Oxygen

The solubility of oxygen in silicon has been investigated by several [9 -14]. For the equilibrium between solid silica and dissolved oxygen in liquid silicon Iguchi and Narushima [12] found

$$\log[\%O] = 0.332 - \frac{4,620}{T} (\pm 0.06) \quad (T: 1693 - 1823 \text{ K}) \quad (4)$$

Ottem [14] gives for the same reaction:

$$\log[\%O] = 1.5205 - \frac{6,975}{T} \quad (T: 1723 - 1923 \text{ K}) \quad (5)$$

The difference in calculated [%O] from these expressions are nearly independent on the temperature and with the values of Iguchi and Narushima [12] about 15 ppm higher than those of Ottem [14]. From the thermochemical data base [16] the standard Gibbs energy for the formation of SiO₂ (crist.) is: $\Delta G^0 = -946,388 + 198 \cdot T$ [J/mole]. By employing equation (3) with the solubility data of Ottem in equation (5) one obtains for the dissolution of oxygen in liquid silicon

$$\frac{1}{2}O_2(g) = \underline{O} \text{ (1 mass\%); } \quad \Delta G_6^0 = -339,663 + 69.9 \times T \text{ [J/mol]} \quad (6)$$

This expression differs slightly from the same given by Ottem who used the data of Rein and Chipman for the standard Gibbs energy of formation of SiO₂.

2.2 Carbon

The solubility of carbon in liquid silicon in equilibrium with SiC(s) was measured among others by both Iguchi and Narushima [12] and Ottem [14]. The differences in results on carbon are more temperature dependent than for oxygen solubilities. At 1450 °C the difference is a factor of about 2 while at 1600 °C the factor is 1.25. Again [14] has measured the lowest values. Using his results where

$$[\%C] = 3,359 \cdot \exp\left(-\frac{22,417}{T}\right) \text{ [mass\%]} \quad (7)$$

and the standard Gibbs energy from [16] for the reaction

$$Si(l) + C(s) = SiC(s); \quad \Delta G_8^0 = -122,870 + 36.8 \times T \text{ [J/mol]} \quad (8)$$

gives for the solution of C(s) to 1 mass% in liquid silicon

$$C(s) = \underline{C} \text{ (1 mass\%); } \quad \Delta G_9^0 = 63,516 - 30.7 \times T \text{ [J/mol]} \quad (9)$$

2.3 Nitrogen

The nitrogen solubility in liquid silicon in equilibrium with Si₃N₄ (s) was investigated by Iguchi and Narushima [12] and they found that

$$\log[\%N] = 2.410 - \frac{9,759}{T} (\pm 0.24) \quad (T: 1723 - 1873 \text{ K}) \quad (10)$$

Extrapolated to the melting point of silicon gives a solubility of 4 ppm mass. This is low compared with the results of Yatsurugi et al. [10] who found the solubility in liquid silicon at the melting point to be 6×10^{18} atoms/cm³ = 55 ppm. The reason for this deviation may be that Yatsurugi et al. used Si₃N₄ powder while Iguchi and Narushima employed a Si₃N₄ crucible. Combining the result of Iguchi and Narushima with standard Gibbs energy data for the formation of solid Si₃N₄, $\Delta G^0 = -877,375 + 408.8 \times T$ [J/mol], taken from [16] gives:

$$\frac{1}{2}\text{N}_2(\text{g}) = \text{N} \text{ (1 mass\%); } \Delta G_{11}^0 = -32,599 + 56.1 \times T \text{ [J/mol]} \quad (11)$$

2.4. Hydrogen

The solubility of hydrogen in iron and iron - silicon alloys has been investigated by several [17- 20]. Mitra and Lange [20] measured the solubility in Fe-Si and other iron based alloys employing Sievert's method [21] for T= 1823 - 1923 K. For pure liquid silicon their results can be transformed to

$$\log[\%H] = 0.0447 - \frac{5,332}{T} \quad (12)$$

which gives 16 ppm at 1873 K. This value lies between the lowest and the highest of the reported solubilities at 1873 K which are 9 ppm [18] and 35 ppm [19] respectively. The standard Gibbs energy for solution of H to 1 mass% will be, using equation (12),

$$\frac{1}{2}\text{H}_2(\text{g}) = \text{H} \text{ (1 mass\%); } \Delta G_{13}^0 = 102,080 - 0.86 \times T \text{ [J/mol]} \quad (13)$$

The Gibbs energies for the elements listed above are compiled in table II together with values for Ca and Al as given by Ottem [14].

TABLE II Change in standard Gibbs energies for solution of elements to 1 mass% solution in Si (l)

Element, <i>i</i>	ΔG_i^0 (J/mol)	ref.
C (s)	63,516 - 30.7 × <i>T</i>	
½H ₂ (g)	102,080 - 0.86 × <i>T</i>	
½N ₂ (g)	- 32,599 + 56.1 × <i>T</i>	
½O ₂ (g)	- 339,663 + 69.9 × <i>T</i>	
Al (l)	- 116,795 + 12.4 × <i>T</i>	[14]
Ca (l)	- 191,272 + 4.1 × <i>T</i>	[14]

3. The influence of Fe on the solubilities in liquid silicon and the interaction coefficient e_{H}^{Fe}

Literature data are scarce concerning the solubility of oxygen, carbon and nitrogen in ferrosilicon with silicon as the main component. For hydrogen more data are available [17 - 20]. Mitra and Lange [20] measured the solubility in alloys containing from 0 - 100 % Si. As for pure silicon, the following relationship can be deduced for FeSi75

$$\log[\%H]_{\text{FeSi75(l)}} = -0.6638 - \frac{4,526}{T} \text{ [mass\%]} \quad (14)$$

We regard FeSi75 as a "low alloy" melt of silicon alloyed with 25 mass% iron. 25% seems to be a high amount of alloying element, however it represents only 14.6 at%. Thus, as an approximation, we only take the first order interaction coefficients into account. For pure silicon the activity of hydrogen is $a_{\text{H}} = [\%H]$ since we then set $f_{\text{H}} = 1$. In FeSi75 the activity $a_{\text{H}} = [\%H]_{\text{FeSi75}} \times f_{\text{H, FeSi75}}$, where for the Henrian activity coefficient $\log f_{\text{H, FeSi75}} \approx e_{\text{H}}^{\text{Fe}} \times [\%Fe]$. The interaction coefficient can

then be calculated by combining equation (12) and (14)

$$[25 \text{ mass\%}] \times e_{\text{H}}^{\text{Fe}} = \log [\% \text{H}]_{\text{Si(l)}} - \log [\% \text{H}]_{\text{FeSi75(l)}} \quad (15)$$

or

$$e_{\text{H}}^{\text{Fe}} = 0.028 - \frac{32.2}{T} \quad (16)$$

which gives $e_{\text{H}}^{\text{Fe}} = 0.011$ at 1600°C .

For the calculation of the interaction coefficients of iron on carbon and oxygen in FeSi the activity of silicon has to be taken into account. From [14] the solubility of C in liquid FeSi75 is deduced to be

$$\log [\% \text{C}]_{\text{FeSi75(l)}} = 3.6 - \frac{10,208}{T} [\text{mass\%}] \quad (17)$$

Combined with equation (7) gives

$$[25 \text{ mass\%}] \times e_{\text{C}}^{\text{Fe}} = \log [\% \text{C}]_{\text{Si(l)}} - \log a_{\text{Si}} - \log [\% \text{C}]_{\text{FeSi75(l)}} \quad (18)$$

or

$$e_{\text{C}}^{\text{Fe}} = 4.5 \times 10^{-4} + \frac{18.88}{T} \quad (19)$$

Shevtsov [15] investigated the solubility of oxygen in FeSi in the range 0 - 88,2% Si. Ottem [14] studied the solubility from 50 to 100% Si and found about 1/10 of the values given by Shevtsov. Both investigated the equilibrium between the liquid alloy and $\text{SiO}_2(\text{s})$. From [14] the solubility of oxygen in FeSi75 can be deduced and is

$$\log [\% \text{O}]_{\text{FeSi75(l)}} = 0.751 - \frac{5,264}{T} [\text{mass\%}] \quad (20)$$

Compared with C and H, the solubility of oxygen in liquid Fe-Si alloys has a maximum at ~80 mass% [14, 15]. The second-order interaction coefficient, r_{O}^{Fe} , can not be neglected and from [14] we have at 1873 K $\varepsilon_{\text{O}}^{\text{Fe}} = -7.75$ which gives $e_{\text{O}}^{\text{Fe}} = -0.015$. How to convert to the interaction coefficient on weight basis is among others given by Lupis in [22]. With the same approach as for carbon, r_{O}^{Fe} is calculated to be 4×10^{-4} . The data are compiled in Table III.

TABLE III Interaction coefficients e_i^{Fe} in iron-rich silicon - iron alloys at 1600°C

Element, <i>i</i>	e_i^{Fe}	r_i^{Fe}
C	0.011	
H	0.011	
N		
O	-0.015	4×10^{-4}

4. Calculated solubility versus measured levels in ferrosilicon.

Solubilities are lowered as the temperature decreases giving secondary inclusions. For this reason it is attempted to compare solubilities at the tapping temperature 1600°C and casting temperature 1450°C . By employing the standard Gibbs energy for solution from Table II combined with thermochemical data from a data base like [16], the solubility of the elements C, H, N and O in pure liquid silicon can be calculated for the temperature of interest. Regarding FeSi75 as a low alloy melt of silicon with 25 mass%, the solubility of C, H, N and O in liquid FeSi can be calculated using the interaction coefficients in Table III together with the Gibbs energies in Table II provided that the activity of Si in liquid FeSi is known.

Table IV gives the calculated solubility figures at 1600 °C and 1450 °C for both liquid silicon and ferrosilicon. In the calculations $a_{\text{Si}} = 0.83$ is used for FeSi75 [23].

For comparison, measured values for commercial FeSi75 are also given. The values for hydrogen, nitrogen and oxygen for this commercial FeSi75 are from an extensive investigation in 1992, where the level of these gases was measured as function of storage time, size and humidity in the surroundings for a wide range of commercial ferroalloys [26]. The carbon levels are typical in production. As seen from Table IV, the measured level of hydrogen and nitrogen corresponds well with the solubility figures. For the case of carbon and oxygen the level varies over a wide range from the solubility level and up to 100 times this level for oxygen. This deviation between the solubility of C and O and measured levels in commercial FeSi75 will be discussed in the following.

TABLE IV: Calculated solubility of C, H, N and O in Si (l) and FeSi75 (l) at 1600 °C and 1450 °C and measured content of the same elements in commercial FeSi75 in ppm mass.

	Carbon		Hydrogen		Nitrogen		Oxygen	
	Si	FeSi75	Si	FeSi75	Si	FeSi75	Si	FeSi75
1600 °C (calc)	213	140	16	8	16	18 ^{*)}	63	90
1450 °C (calc)	75	47	9	5	6	6 ^{*)}	30	50
Commercial FeSi75 [28]		75 - 1000		3 - 5		4 - 45		60 - 3000

^{*)} e_{N}^{Fe} has been taken equal to zero

FILTER SAMPLER FOR MEASURING INCLUSIONS IN FESI AND SI

Since ferrosilicon (75 mass% Si) typically has a tapping temperature between 1500 - 1700 °C compared with 700 °C for aluminium, it was necessary to develop sampling equipment that would withstand temperatures up to 1700 °C. The trials were carried out at Elkem Thamshavn, on metal tapped from furnace number 2. This furnace has an annual capacity of 43,000 mt. The metal is tapped into 3 m³ ladles. Figure 1 shows the experimental arrangement including the tapping ladle, and an enlarged drawing of the filter sampler to the right. The essential parts of this device are a filter holder attached to a canister where the filtered metal is collected. All parts including the filter are made of graphite as shown in figure 2. A more thorough description of the equipment is given in [26]. After tapping the ladle was transported to the sampling station. To heat the sampler to the temperature of the melt the sampler was first held in the melt for ~30 seconds. To avoid contact between melt and filter before the filter was preheated, nitrogen was blown down through the canister, through the channels and up the filter. When a sufficiently high

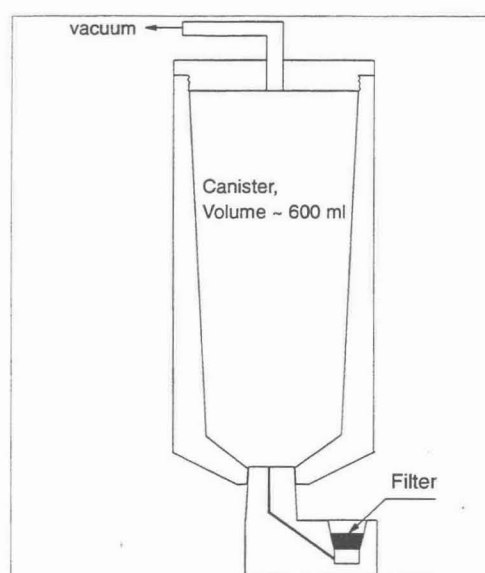


FIG. 2: Detail from figure 1 showing the Graphite Filter Sampler

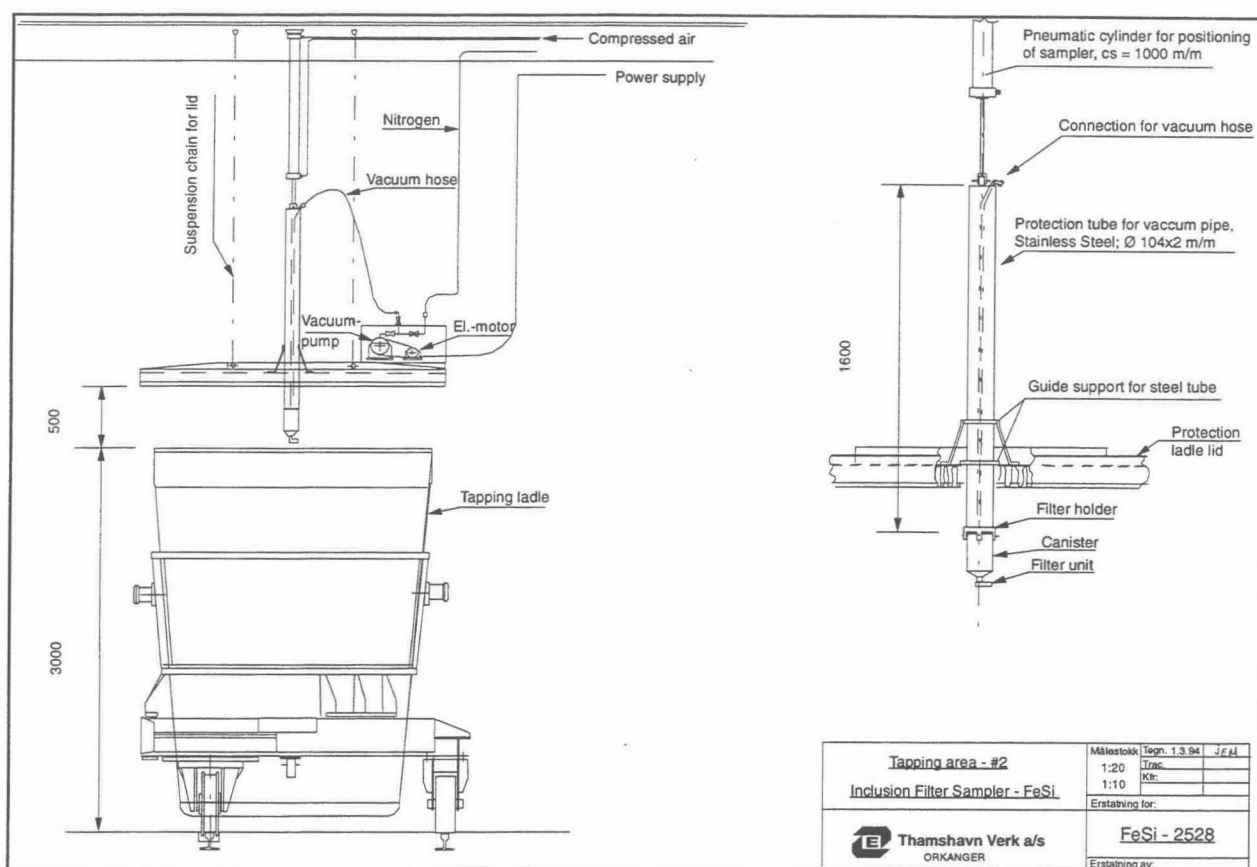


FIG. 1: Experimental layout for vacuum filter sampling of ferrosilicon at Elkem Thamshavn

temperature was attained, the valve was opened to the vacuum system and the nitrogen flow was shut off. Inclusions were collected in the filter for about 2 minutes. As the sampler was taken out of the melt, the vacuum valve was closed. The sampler was then cooled with the use of a blaster connected to the nitrogen source. The samples were taken from production runs. Thus the chemical composition of sampled metal, the amount of slag, tapping time, etc. may vary somewhat from one run to the other. In Table V some parameters that could be significant for the evaluation of the results are shown. The trial number corresponds to the melting plant tapping number.

TABLE V: Tapping data for the trials

Trial no	Duration of tapping	Temp. before sampling	Tapping weight	"Slag"	Tapping analysis		FeSi- Fines added	Bottom gas stirring
	(min)	(°C)	(kg)	(kg)	Al (%)	Ca (%)		
623	100	1554	9100	780	1.29	0.14	450	Air/O ₂ -mix
624	65	1610	8870	30	1.27	0.14		
141	65	1511	9210	140	1.32	0.20		
220	130	1492	10230	360	1.09	0.13		
616	75	1565	10470	620	1.18	0.18	10 Nm ³ air	10 Nm ³ air
617	55	1644	9560	300	1.18	0.18		
716	50	1610	7770	240	1.23	0.19		
718	80	1550	9040	250	1.16	0.19		
								34 Nm ³ air/O ₂

EXPERIMENTAL RESULTS AND DISCUSSION

The samples were prepared for metallographical examination. Microprobe (EPMA) analysis has shown that the particles are SiC and mixed oxides. The analysed oxides correspond to typical furnace slag and are probably not precipitates or endogenous inclusions.

The automatic image analysis and the calculation of the number size distribution are described in detail in [4, 24-26]. Here it is pointed out that 1) the particles are assumed to be spherical and 2) the size distribution per volume is calculated from size distribution per area in accordance with Saltykov's theory.

$$N_v(d_i) = \frac{1}{\Delta} [\alpha_{ij} \times N_A(d_i) - \sum_{k=1+1}^1 \alpha_{i,k} \times N_A(d_k)] \quad (21)$$

where Δ is the width of the size classes and the α_{ij} are the weights due to Saltykov.

The calculation software program used, gives the concentration of inclusions in the melt and the mean particle diameter in addition to the number size distribution. Table VI presents the volume fraction of inclusion, the mean particle diameter and the amount of metal filtered. The number size distributions are presented graphically in figure 3. The inclusion number size distribution for the samples is roughly a decreasing exponential function as often obtained [27]. The number size distribution curves in figures 3 show that the ferrosilicon investigated contains a large number of small inclusions. There are small amounts of inclusions greater than 15 μm . Whether this applies only to the ferrosilicon investigated or is a more general problem is not possible to say. But the large variation in the oxygen and carbon level and the high amount of oxygen

and carbon that may be found in commercial ferrosilicon compared with the solubility figures, see table IV, can be due to a variable content of inclusions in ferrosilicon melts. The 3 heats with bottom gas stirring have a lower concentration of inclusions. Trial 623 has about half the amount of particles below 5 μm compared with trial 624. Trial number 718, with bottom gas stirring, has only $\frac{1}{4}$ the number of inclusions below 5 μm compared with 716. The reason for this effect can be that particles are trapped in the slag produced by the reaction between oxygen and aluminium and calcium or they may be removed due to turbulent removal to the lining. This subject is treated in chapter 5 in [27]. Figure 4 shows a micrograph with SiC particles trapped in slag.

TABLE VI: Inclusion concentration and mean particle diameter

Trial no	Amount filtered (gram)	Mass-flow (g/min)	Inclusion conc. on volume (ppm)	Mean particle diameter (μm)
623*	494	n.a.	145	3.38
624	389	292	479	3.75
141	384	192	190	1.85
220	782	313	3014	4.07
616	1524	762	222	4.23
617*	1139	455	201	3.64
716	1498	749	215	3.28
718*	1055	422	79	3.71

*With bottom stirring

CONCLUSION

The standard Gibbs energy change for the solution of Al, Ca, C, H, N, and O in liquid Si has been calculated based on solubility data from the literature review. The solubilities of C, H, N, and O in liquid FeSi75 are calculated regarding FeSi75 as a low alloy melt of silicon with 25 mass% Fe (14 at%) and employing the interaction coefficients given in Table III.

Ferrosilicon with 75% Si contains a high number of small inclusions, consisting of SiC and mixed oxides. These inclusions, which can be measured employing the filter sampling technique developed

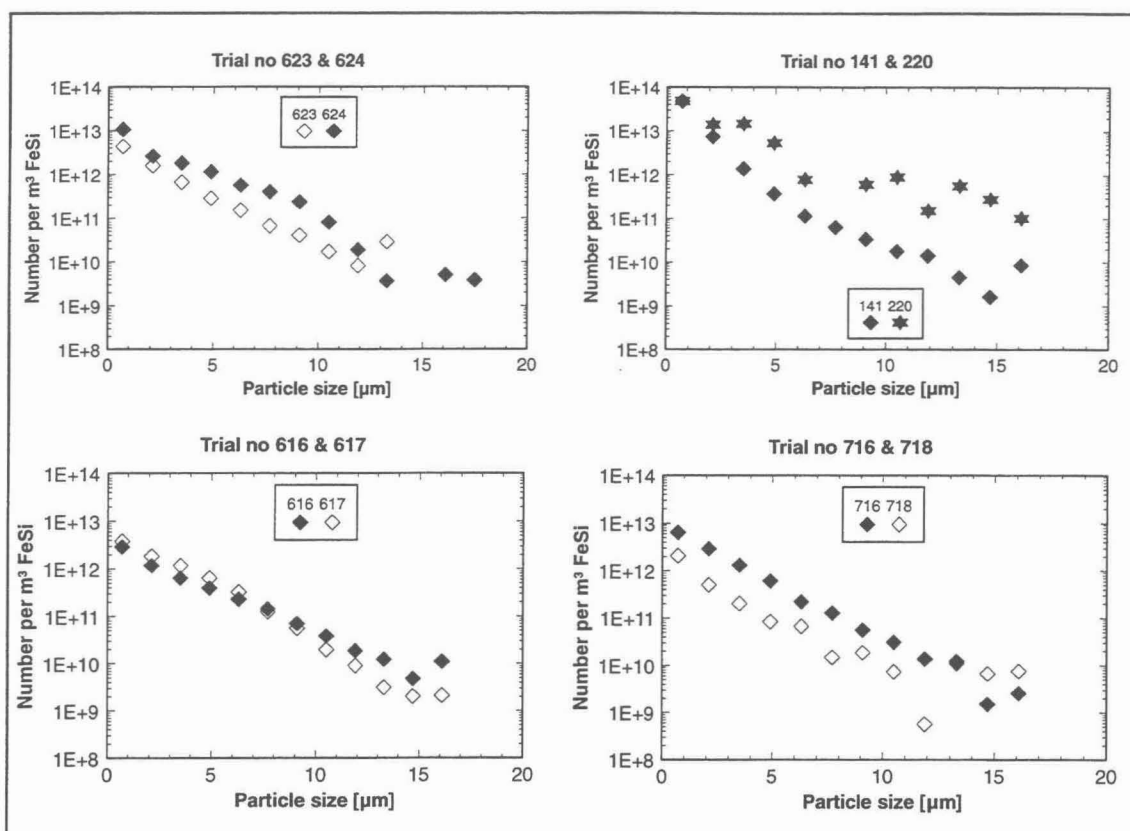


FIG. 3: Inclusion number size distribution in FeSi75 from production run at Elkem Thamshavn; Filled points indicate heats with no stirring. Empty points are with bottom stirring.

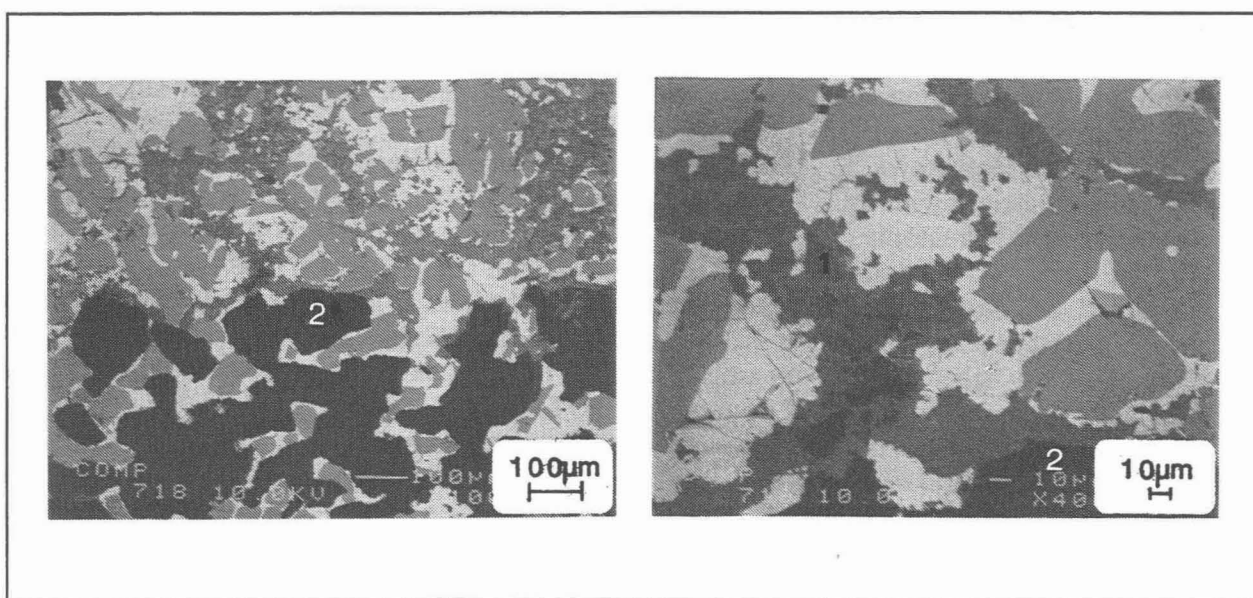


FIG. 4: Micrographs (EPMA) of inclusions in FeSi75. Trial 718. Flow direction from left to right. ① SiC-particles trapped in slag. ② Filter. (Right picture is a segment of the picture to the left.)

here, is probably the main reason for the high level of total oxygen and carbon found in commercial ferrosilicon. By employing bottom gas stirring the number of inclusions is reduced and the cleanliness of the alloy improved.

ACKNOWLEDGEMENTS

The authors wish to acknowledge Mr Leif Kristiansen at Elkem Thamshavn for his assistance in the experiments. Professor J.Kr. Tuset is to be thanked for his valuable advice when compiling the solubility data. Dr.ing Frede Frisvold is acknowledged for his assistance with the calculation of the number size distributions.

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