

STUDY OF SiMn PRODUCTION IN PILOT SCALE EXPERIMENTS – COMPARING CARAJAS SINTER AND ASSMANG ORE

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ABSTRACT

Production of SiMn has been studied by 3 experiments in a 150 kW pilot scale submerged arc (SAF) furnace. Two of the experiments were with sinter from Carajas ore as raw material while the third experiment was with a 40/60 mixture of sinter from Carajas ore and ore from Assmang. Alloy produced in the two first experiments had a lower Si- content than alloy from the experiment where Assmang ore was mixed with Carajas sinter. Measurements of melting properties of the Mn-sources showed that the sinter melted at a lower temperature than the Assmang ore. This is believed to contribute to the lower % Si in alloy produced by sinter.

1. BACKGROUND

In 2006 the global production of SiMn was 6.9 million tons [1] with steel production as the primary user. Several different grades are produced; for standard SiMn the analysis are in the range 65-68 % Mn, 1.5-3 % C and 12.5-21% Si. The major part of SiMn is produced in submerged arc furnaces (SAF) by carbothermic reduction of oxidic raw materials. Manganese ores and HCFeMn slags are the main Mn sources and quartz the main Si-source. Fluxes as dolomite and limestone are added to adjust the properties of the final slag. Several carbon sources are used as reductant, coke is most common and the only source used in Norway. The process and reactions are extensively studied and described by Olsen et al [2].

During production of silicomanganese alloys, the submerged arc furnace can be divided into two main reaction zones [2]. Their existences are confirmed by excavation of industrial furnace of Mn-alloys [3]. In the upper part of the furnace, in the prereluction zone the raw materials are solid and reductions take place by gas-solid reactions. In the lower part of the furnace, in the coke bed, slag and metal are liquid. Here heat is generated by ohmic resistance in solid coke. Most of the supplied energy is consumed here, and the final reduction of MnO to Mn and of SiO₂ to Si takes place in this zone by the following main reactions.



At the top of the coke bed the initial slag is formed by melting and reduction of the Mn-sources and dissolution of the fluxes. When the viscosity of the slag is low enough, it will drain into the coke bed. The temperature where this is achieved defines the temperature at the top of the coke bed and determines the temperature in the coke bed [4]. Earlier pilot experiments have shown that most of the reduction takes place at the top of the coke bed. Melting properties of the ores is thus

believed to affect the temperature in the zone and by this be of importance for furnace performance. This effect has been seen industrially in HCFeCr production as a correlation between melting properties of ores and Si-content in alloy [5]. In charge mixtures addition of quartz will lower the melting temperature with around 100°C while addition of dolomite will increase the melting temperatures with around 100°C [6]. The total charge mixture must be considered when melting properties are discussed.

Comparison of melting properties of ores and agglomerates have shown that acidic Mn-ores have higher melting temperatures than sinters made of these ores and that CVRD ores have relatively low melting temperatures compared to other ores [4, 8, 9]. Under otherwise equal conditions, this is believed to give lower temperatures in the coke bed zone and affect the reduction of oxides and the composition of slag and alloy. The effect is discussed and not clear. Sinter from Carajas Mn-ore (CVRD-ore) was in 2006 the main Mn-source for Vale Manganese Norway (now Glencore). In this period SiMn-alloy production at Vale gave lower % Si in the alloy and higher % MnO in the slag than SiMn production by comparable producers. In order to identify possible reasons for this difference, melting temperatures for various Mn-sources were measured and 3 pilot scale experiments were run by Sintef. The main objective of these experiments was to see

1. The difference of substituting Carajas sinter (CVRD sinter) with Assmang ore.
2. The effect of increased initial coke bed size on production with Carajas sinter.

Some of the results are included in other publications [9, 10]. Here the 3 pilot experiments are compared.

2. RAW MATERIALS

In addition to the Mn-sources, CVRD sinter and Assmang ore, quartz was used as Si-source, coke from Corus as reductant and dolomite as flux. Chemical compositions of the raw materials are shown in table 1.

Table 1: Chemical composition of raw materials

Name	H ₂ O	Mn	OMn	Fe	SiO ₂	CaO	MgO	Al ₂ O ₃	K ₂ O	C
CVRD sinter	1.0	49.0	17.0	7.9	7.2	0.7	0.4	10.5	1.4	
Assmang ore	2.1	47.0	25.0	10.0	5.0	7.3	0.5	0.2	0.0	
Corus coke	9.6			0.4	6.5	0.4		3.4	0.1	83.1
Quartz	1.0			0.1	98.8			0.7		
Dolomite	2.0			0.1		30.1	20.4	0.1		

Melting temperatures for the Mn sources presented in table 2 showed that CVRD sinter melts at lower temperature than Assmang ore.

Table 2: Melting temperatures for selected Mn-sources

Element	Assmang ore	CVRD sinter	Comilog ore	HCFeMn slag
Temperature, °C, of initial softening of Mn sources	1450 °C	1350 °C	1490 °C	1220 °C
Temperature, °C, of final melting of Mn sources	1510 °C	1410 °C	1540 °C	1230 °C

The measured samples were split out from the same batch that was used in the pilot experiments. Comilog ore and HCFeMn slag were measured for comparison. The temperatures

were measured in a sessile drop furnace and measurement method and the results from these investigations are published earlier [4]. Later investigations of Assmang ore and CVRD sinter confirms the higher melting temperatures for Assmang ore compared to CVRD sinter [10,11]. The higher melting temperatures for Assmang ore compared to CVRD ore can be explained as a result of the higher basicity of Assmang ore as discussed by Visser [10]. Tangstad [8] has earlier shown that CVRD sinter has lower melting temperatures than CVRD ore and contributed this to the smaller primary grain size of sinter. The implication of lower melting temperatures for sinters is not fully known. As described in the introduction it is expected to give lower temperatures at the top of the cokebed and make it more difficult to achieve high Si-content in the alloy.

3. PILOT EXPERIMENTS

In all three experiments SiMn was produced in a 150 kW pilot scale furnace at SINTEF /NTNU. The experiments were run with a top and bottom electrode. The setup is shown in figure 1. The lining is designed with respect to an excavation technique developed by B. Heiland at NTNU /Eramet where it is possible to make casted cross sections of the furnace.

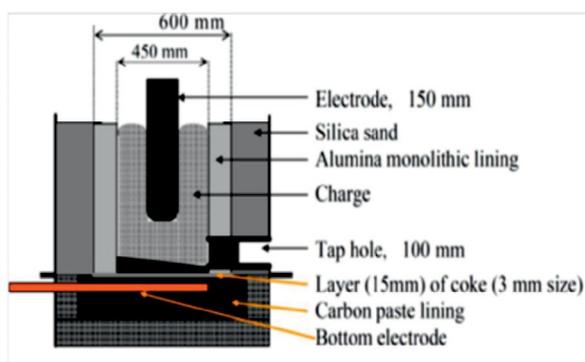


Figure 1: Sketch of pilot scale furnace

The SiMn charge mixtures used in the experiments are shown in table 3. The target product composition for the experiments was an alloy with 16.5 % Si and a slag with 15 % Mn, 38% SiO₂

and an R ratio of 0.98 where R is defined as
$$R = \frac{MgO + CaO}{Al_2O_3}$$
 In the experiments with Assmang ore, the Mn-sources were a mixture of 60 % Assmang ore and 40 % sinter. Since Assmang is a basic ore, less dolomite is needed when this is used. Stoichiometric amount of coke was added. In this type of pilot experiments, each batch of charge consists of 50 kg Mn-sources.

In the experiments a starting coke bed was first built in the furnace. This was preheated, and then the raw materials were charged into the furnace. In order to reach high enough temperature for silica reduction before quartz is added, the first charge was for HCFemn production. This is critical to avoid a quartz layer in the coke bed that will disturb current and energy distribution in the furnace. The furnace was charged continuously to always keep it filled with charge. The electrode tip was set 20 cm above the bottom of the furnace when the experiment started and the target was to keep it at this position during the whole experiment. Furnace load was kept at 150 kW by changing transformer tap. The electrical parameters are logged and can be analysed during the experiment. When large deviations from planned values or large instability in electrical parameters are observed, the electrical operation can be corrected. Operational differences due to this may conceal differences between the experiments and make comparison of experiments difficult. The furnace was tapped each 80 kWh and had 8 tappings in experiment 1, 9 in exp. 2, and 7 in exp. 3. The

furnace was shut down 50 kWh after last tapping. When the experiment was finished and the furnace had cooled, it was filled with epoxy. After hardening a cross section plate through the electrode centre was cut out from the furnace. Occurrence of the different zones in the furnace were identified and described. From experiment 1 and 2, samples were taken out from the plate and investigated by microprobe.

Table 3: Charge mixtures used in the pilot experiments

Experiment	CVRD 3 kg coke bed	CVRD 5 kg coke bed	CVRD + Assmang
Carajas sinter kg	50	50	22.5
Assmang ore kg	0	0	17.5
Coke kg	13	14	16
Quartz kg	18	18	14
Dolomite kg	10	10	1
Starting coke bed kg	3	5	5
Start charges with low quartz	3	4	4
SiMn charges, number	6	6	5

The first two experiments were run by Sintef together with personnel from Vale, while the last was run as student work at NTNU with some involvement from Sintef. The change in operational personnel might have affected the control of the furnace and the electrical parameters. The experiments were run for approximately 8 hours. This is short enough to avoid electrode wear so electrode tip position can be known, and at the same time estimated from earlier experiments to be long enough to achieve stable conditions in the cokebed. In industrial operation electrode position is adjusted to keep a constant resistance or current. When the position of the electrode tip is changed, size and shape of the coke bed and consequently the melting and reduction conditions will change. The furnace then goes through a transition period before it stabilise again. In the experiments presented here electrode position was kept constant. Measured resistance will then vary with conditions in the coke bed and can indicate how different raw materials or other parameters affect conditions in the reduction zone.

4. RESULTS PILOT EXPERIMENTS WITH CVRD SINTER AND ASSMANG ORE

Pilot experiments of the type run here are designed to give relative differences between parameters. The study of furnace interior will in addition give insight in mechanisms and distribution of reaction zones that can explain the observed differences. The absolute values for product quality or electrical conditions cannot be directly compared with results from industrial furnaces. This is due to the short operational time and the large surface effects in the relatively small furnace.

A good furnace performance is achieved when the operation and the electric parameters are stable, the yield is high and the produced alloy meets the quality requirements. This will give a stable and evenly distributed flow of off-gases from furnace top and continuously descending of charge materials that both can easily be seen in the pilot furnace. A stable resistance will indicate a stable coke bed with the possibility for good melting conditions. The resistance was lower and the electrical parameters more stable and easier to control when Assmang ore was included in the charge mixture. The average values for the period with stable SiMn production are seen in table 4, while their variation during the experiments are shown in figure 3, 4. Average load was in all the experiments lower than the target of 150 kW. When the resistance was too high or too low, it was

not possible to achieve 150 kW by controlling the voltage. Variations in load seen in figure 3 are mainly a result of variations in resistance seen in figure 4.

Table 4: Electrical parameters SiMn production

	CVRD sinter, 3 kg coke bed	CVRD sinter, 5 kg coke bed	CVRD sinter + Assmang ore
Load, kW	140.5	130.8	148.9
Resistance, Ωm	22.3	26.1	6.9
Current, kA	2.7	2.3	4.7
Voltage, V	54.3	56.9	32.4

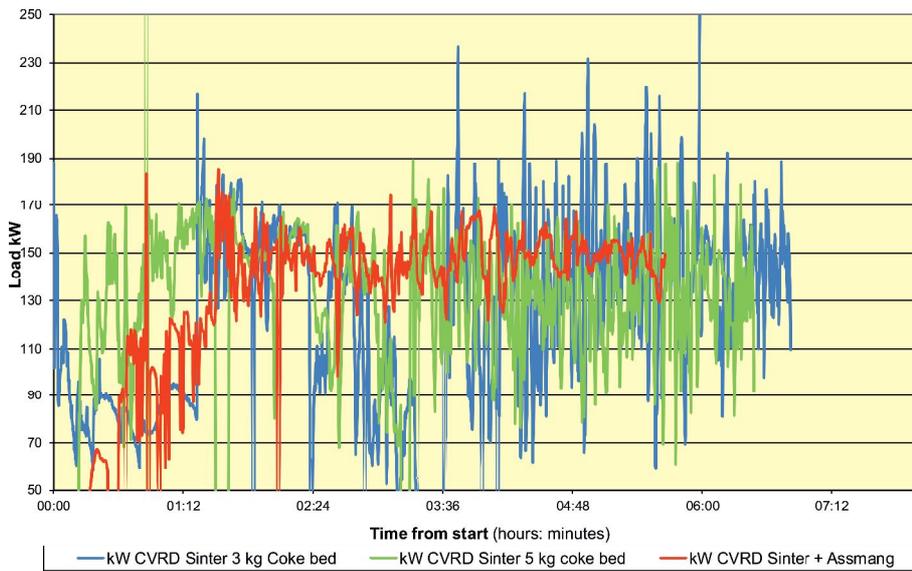


Figure 2: Load, kW in the pilot experiments

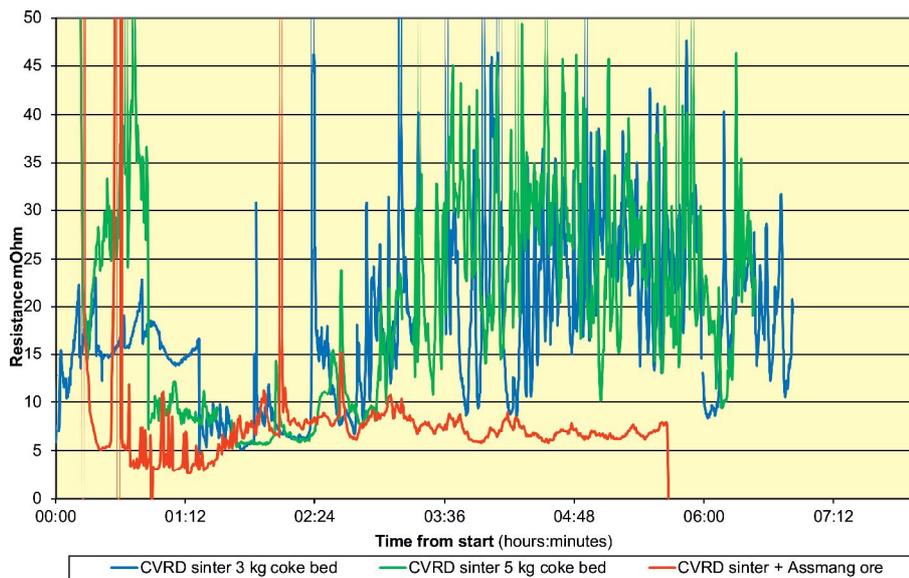


Figure 3 Resistance, m Ω in pilot experiments

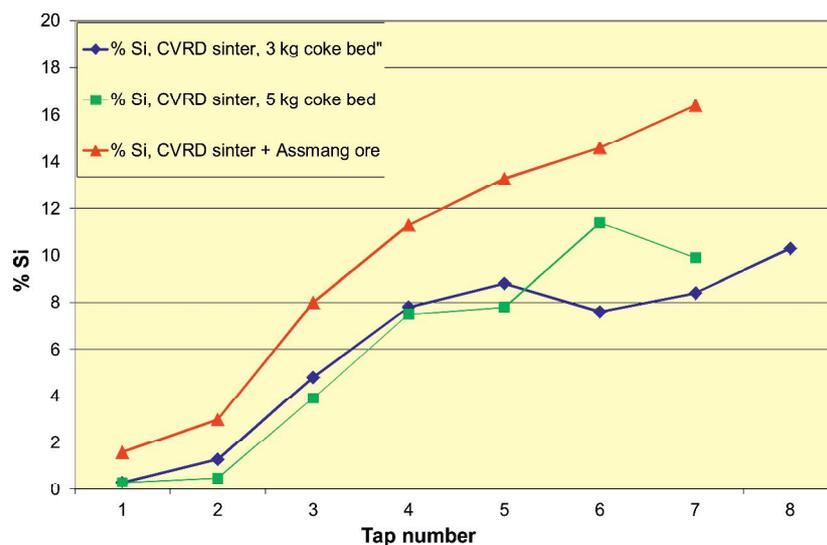


Figure 4: %Si in tapped alloy in the three experiments

During some of the tappings, electrical connection was lost, and the electrode had to be lowered. This was especially the case in the experiments with small, 3 kg coke bed. When 60 % of the CVRD sinter was replaced with Assmang ore, both resistance and load was more stable and an average load of 149 kW was reached.

In the two experiments with CVRD sinter, furnace operation seemed unstable. The gas flow from the charge top was erratic with some blows, and the flow of charge materials varied both over furnace area and with time. Some collapses of charge were observed. When 60 % of CVRD sinter in the charge was replaced with Assmang ore, the furnace operation became much more calm and stable with even gas flow and continuous descending of charge materials.

Composition of tapping with highest % Si for all 3 experiments is shown in table 5 and variation in % Si in tapped alloy in figure 5. % Si in the alloy was higher with Assmang ore in the charge than when only CVRD sinter was used.

Table 5: Chemical analysis of tap with highest % Si

	CVRD sinter, 3 kg coke bed	CVRD sinter, 5 kg coke bed	CVRD sinter + Assmang ore
% Si	10.3	11.4	16.4
% Mn in alloy	66.8	69.5	66.7
% MnO in slag	26.4	22.1	35.6
% SiO ₂	35.1	36.7	35.0
% CaO	8.4	9.0	12.5
% MgO	6.2	6.6	3.8
% Al ₂ O ₃	22.5	24.1	14.2

Low MnO content is a target for industrial production. In pilot experiments it is taken as an indication of good melting and reduction in the coke bed and is expected to correlate with high % Si in the alloy. This correlation is not seen here. The high MnO content in slag from experiments with Assmang ore might be caused by metal droplets in the slag. Compared with industrial operation all the slags have a high MnO content. The tapped slag is as discussed by Tangstad [12] a mixture of materials from the high temperature reduction zone and less reduced materials mixed in from the

sides. In pilot experiments, relatively more material is mixed in from the sides and higher MnO is expected.

The coke bed could be seen in the casted cross sections of the furnaces, from all three experiments as shown by the idealised sketch in figure 5 and the picture of a plate in figure 6.

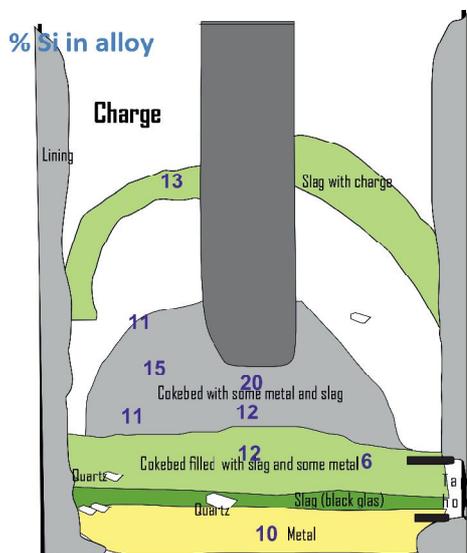


Figure 5: Cross section experiment with CVRD sinter, 5 kg coke bed

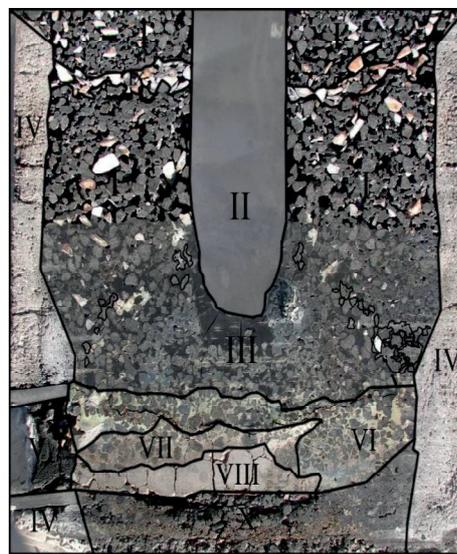


Figure 6: Cross section experiment with CVRD sinter and Assmang ore

Amount of metal and slag increased downwards in the coke bed. In the two experiments with only CVRD sinter, unreacted quartz was found at the bottom of the furnace while this was not seen in the experiment with Assmang ore. Unreacted quartz found in the coke bed in the experiments with CVRD sinter can be a result of different raw materials, giving a lower temperature, or of differences in operation. In the two experiments with only CVRD sinter, samples were taken out from the cross section of the furnace and investigated by Microprobe. This confirmed the observation from other pilot scale experiments [11, 13, 14] that most of the reduction of both MnO and Si takes place at the top of the coke bed. Both in the published and in the current experiments, % Si in alloy was higher in the middle than on the top of the coke bed. No samples were investigated from the experiment with Assmang ore.

5. EFFECT OF USING ASSMANG ORE IN THE CHARGE MIXTURE

A comparison of the results in this series of experiments shows insignificant effect of increasing initial coke bed from 3 to 5 kg while replacing 60 % of CVRD sinter in the charge with Assmang ore has a large effect. Use of Assmang ores gave higher % Si in the tapped alloy than when CVRD sinter was the only Mn-source, 16.4 % versus 11.4 % Si. Melting temperatures for Assmang ores are around 100°C higher than for CVRD sinter. The higher melting temperature is expected to give higher temperature at the top of and in the coke bed and a by this better conditions for reduction and higher Si in the alloy. This is in accordance with what is seen in the pilot experiments.

With use of Assmang ore, electrical conditions in the furnace was more stable than with only CVRD sinter in the charge. It was easier to keep the target load of 150 kW and the furnace resistance was much more stable and lower, around 1/3 of the resistance with use of only CVRD sinter. In the experiments with only CVRD sinter, the coke bed contained pieces of unreacted

quartz as shown in figure 6. Too much unreacted quartz in the coke bed will affect current distribution in the furnace and give higher resistance, a wider coke bed with lower energy density and poorer reduction. This will result in lower Si in the alloy. The quartz pieces in the coke bed can be a result of low temperature in the coke bed due to low melting temperature of the ore. But they can also result from the operation of the furnace during transition from HCFeMn to SiMn charge. The effect of initial slag composition on dissolution of quartz can also be of importance. This varies with ore type.

A smaller initial coke bed led to problems with keeping the electrical contact and current flow in the furnace and more periods when the electrode must be lowered. An electrode tip position of 20 cm was too much for this coke bed size. Except for this, the experiment was similar to the experiment with 5 kg coke bed.

The experiments in this investigation indicate that Assmang ore in the charge will give higher Si in the alloy than a charge with only CVRD sinter. Other causes for the difference in Si-content cannot be excluded from only three experiments.

6. DISCUSSION

Several SiMn pilot experiments have been run at Sintef. Many of these are a part of direct projects for customers and are confidential. Results from a series of experiments with different ores have been published by Tangstad [11], and a series of experiments with different reductants have been published by Monsen [12, 13]. They are in table 6 compared with the experiments in this investigation. In SiMn production one of the main objectives is to obtain high % Si in the alloy. Achieved values, written by red in the table vary from 10.3 % to 18.9 % Si. Several possible explanations for the differences have been proposed. The experiments in the different series cannot be directly compared since they are designed to reveal relative differences within the series.

Comparison with other pilot experiments can indicate if the observed higher Si content with use of Assmang ore is significant. Electrical parameters vary a lot between the experiments. If we compare only the experiments with coke, the two experiments with CVRD sinter have highest voltage and resistance and lowest current. Since the experiment with Assmang ore has the opposite characteristic this is not believed to be caused by different operational procedure for the Vale series of experiment, but to be a result of properties of CVRD sinter. The Vale experiments have values at the same level as the other experiments also for MnO and SiO₂ in the slag.

A good furnace performance is achieved when the furnace operates with a high load and stable electric parameters, the yield is high and the produced alloy meets the quality requirements. Reduction of MnO and SiO₂ takes place in the coke bed. Good reduction requires high energy density and high temperatures in the coke bed, higher for Si than for MnO. In addition chemical composition of the slag must be suitable. Use of Mn-sources with higher melting temperatures is expected to give higher temperature in the coke bed. Energy density in the coke bed is affected by size of the coke bed in addition to type and particle size of the reductant [13, 15]. Unreacted quartz pieces in the coke bed are expected to give higher resistance and in addition more electrical current will flow around the quartz and give a larger coke bed with lower temperature in the reduction zone. Best reduction is normally achieved with a stable coke bed with little variation in load and resistance.

The main conclusion from the experiments by Tangstad [11] was that since HC-slag has low melting temperatures, use of HC-slag made it difficult to obtain high Si in the alloy. Both the experiments by Monsen and the experiments in this series seem to contradict this. In experiment BM 1, 18.8 % Si was achieved with HC-slag. But it was only 30 % HC-slag in the charge and this might be too little to have a negative effect. In experiment Vale 1 and 2, % Si was low, although

The data in table 6 also show that for the experiments with high resistance (quartz in the coke bed), % Si in the alloy increases with increasing % SiO₂ in the slag while for experiments with low resistance (no quartz in the coke bed), % Si in the alloy seems to in average decrease with increasing amount of Mn-sources with low melting temperatures, as HC-slag and CVRD sinter. The main question is then how to avoid unreacted quartz in the coke bed, and how is this affected by melting temperatures for the Mn-sources. This has not been a part of this study. Both to confirm the relation between resistance and quartz in coke bed and to understand the mechanism behind are relevant topics for further work to understand SiMn process. The industrial experience that a Si content of about 20% can be achieved with high contents of HC slag in the charge shows that it is possible to produce SiMn with high Si from Mn-sources with low melting temperatures.

Based on this discussion, the observations in the experiments with ore from Vale are comparable with other pilot experiments. A correlation between low % Si in alloy and lower melting temperatures for the Mn-sources are indicated from earlier experiments. The obtained higher % Si in alloy when 60 % of CVRD sinter was replaced with Assmang ore is believed to be a result of higher melting temperatures for Assmang ore.

7. CONCLUSION

In these pilot experiments, % Si in tapped alloy was higher in a charge with 60 % Assmang ore and 40 % CVRD sinter than in a charge with only CVRD sinter. This is believed to be a result of the measured lower melting temperatures for CVRD sinter.

8. ACKNOWLEDGEMEN

Vale Manganese Norway, now owned by Glencore are acknowledged for allowing these results to be published.

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there was no HC-slag in the charge. But the CVRD sinter has low melting properties and might have the same effect as the sinter.

Table 6: Comparison of three series of pilot experiments (Vale from this work, MT by Tangstad [11], BM by Monsen [12, 13])

	Vale 1	Vale 2	Vale 3	MT A	MT B	MT C	MT D	MT E	MT F	BM 1	BM 2	BM 3	BM 4	BM 5	BM 6
Mn sources															
(as % of Mn Sources)															
CVRD sinter %	100	100	40												
Assmang ore %	100	100	60				50	40	70						
Comilog ore %					50	32				70	70	70	70	70	70
BHP ore %						18		60							
Temco sinter %				40											
Mamatwan %				60											
HC-slag %					50	50	50		30	30	30	30	30	30	30
Other charge materials															
(as kg pr kg Mn sources)															
Quartz	0.36	0.36	0.28	0.40	0.21	0.26	0.28	0.36	0.39	0.26	0.26	0.26	0.26	0.26	0.27
Dolomite	0.20	0.20	0.02			0.05	0.09	0.10	0.09						
Limestone				0.08	0.10	0.01		0.04							
Carbon															
Start coke bed kg	3	5	5	-	-	-	-	-	-	5	3	5	5	3	5
Electrode position, cm	20	20	20	10	13	8	5	5	8	20	20	20	15	15	15
Metallurgical coke %	100	100	100	100	100	100	100	100	100	100				54	54
Other reductants %											100	100	100	46	46
Electrical prameters															
Load, kW	141	131	149	100	125	100	104	150	82	152	146	138	151	148	155
Resistance, mΩ	22	26	7	17		8	11	7	26	10	17	36	36	35	15
Current, kA	2.7	2.3	4.7	2.5		3.5	3.4	4.5	2.1	3.9	3.0	2.1	2.1	2.1	3.4
Voltage, V	54	57	32	42	30	28	31	32	40	39	50	67	72	71	48
Tap analysis best tap															
% Si in alloy	10.3	11.4	16.4	18.9	15.7	16.3	14.8	18.1	14.7	18.8*	17.3*	11.4*	13.5*	12.9*	16.6*
%MnO in slag	26.4	22.1	35.6	21.9	5.9	14.5	17.7	18.5	23.1	25.8*	21.4*	22*	17.4*	19*	29.9*
% SiO ₂ in slag	35.1	36.7	35.0	41.3	41.1	43	43.6	44.6	46.8	39.8*	40.3*	42.1*	37.4*	39.2*	37.5*
Slag/metal	2.4	1.3	0.4							0.6	1.1	0.9	0.8	1.2	1.2*

In table 6 no clear correlation between resistance and % Si in alloy can be seen. But unreacted quartz pieces in the coke bed in some of the experiments might hide some correlations. If we assume that all experiments with high resistance have quartz in the coke bed, figure 7 shows that that to achieve high % Si in the alloy, there must be no unreacted quartz in the coke bed.

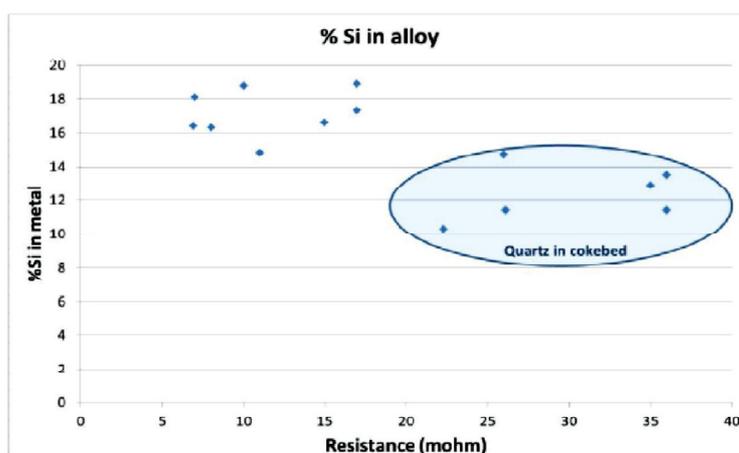


Figure 7: Resistance and % Si in pilot experiments

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