EVALUATION OF NEW POSSIBILITIES OF UTILIZATION OF MANGANESE ORE AND IRON ORE IN THE DIRECT AND SMELTING REDUCTION PROCESSES[1]

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Abstract

The objective of this work is to evaluate new possibilities to utilize Mn ore and Fe ore in direct and smelting reduction processes as a pre-material, a kind of Mn containing sponge iron, for the production of high Mn steels. This includes the use of briquettes, fine ores and the injection of Mn ore into the melt pool as well as the smelting of Mn-ore-Al-briquettes. During the briquette reduction a separation of metal and slag phase occurs at higher temperatures. The reducibility of mixtures of both Fe and Mn ore were analyzed using a micro analyzer and x-ray diffraction.

Key words: Manganese ore; Iron ore; Combined reduction; High manganese steel

AVALIAÇÃO DE NOVAS POSSIBILIDADES DE USO DE MINÉRIO DE MAGANÊS E MINÉRIO DE FERRO NOS PROCESSOS DE REDUÇÃO DIRETA E FUSÃO

Resumo

O objetivo deste trabalho é avaliar novas possibilidades de uso de minério de Mn e minério de Fe nos processos de redução direta e fusão como um pré-material, um tipo de ferro esponja contendo Mn, para a produção de aço com alto teor de Mn. Isto incluí a utilização de briquetes, finos de minério, e injeção de minério de Mn em banho metálico e assim como a fusão dos briquestes compostos de minério de Mn e Al metálico. Durante a redução de briquetes em temperaturas mais elevadas ocorre a separação da fase metálica e da escória. A reducibilidade de misturas de ambos minérios (Fe e Mn) foi analisada com uso de micro sonda e difração de raio-x.

Palavras-chave: Minério de manganês; Minério de ferro; Redução combinada; Aço com alto teor de manganês

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1 INTRODUCTION

More than 90 % of the Mn ore mined is used in the steel industry. The most usual way in which Mn is added to steel is as ferromanganese (FeMn). In the production of regular carbon steel grades, high carbon containing FeMn and silicomanganese are used. However, in special low carbon steels, medium-carbon FeMn, low-carbon FeMn or electrolytic Mn are used. A steel producer has to weigh up the additional costs of the refined Mn alloys against the extra time in de-carburization processes. Manganese occupies a special place among all alloying elements as it is present in almost all types of steel and cast iron. Mn is also used for the production of less expensive austenitic grades of stainless steel by replacing Ni. Mn is usually added after the converter process as FeMn. It is applied to the steel melt to alloy and for combined de-oxidation with silicon. Therefore, the addition of a less expensive form of Mn is desirable during steelmaking to increase Mn content in steel. These factors are behind a search for alternative technologies for adding Mn during steelmaking processes. FeMn is a relatively expensive substance since it is usually produced in a submerged smelting process using electrical energy, or in a FeMn blast furnace using coke. Coal is a cheaper reductant than coke but causes problems by hydrocarbons and tar compounds in the furnace effluent system. The losses of Mn at various stages of the metallurgical cycle and the use of Mn ore with low P-content have to be considered.

Steel grades with higher Mn contents are in development and stronger demand for Mn and Fe bearing raw materials is expected. The global crude steel production grew 7.4 % to 1.32 billion tons surpassing the previous year's record. The global consumption of Mn ferroalloys continues to vary significantly from region to region due to different steel production processes, the quality of the raw materials used such as Fe ore grades and types of steel products produced. However, in 2007 the world annual production of total Mn alloys reached 13.1 million tons and the unit consumption of Mn remains approximately 1 wt. % of steel produced [1].

Steels with a Mn content of 15 to 30 wt. % have a high ductility and an enormous hardening behaviour during plastic forming due to their microstructure. Its use in the car manufacturing industry offers remarkable improvement potential in the area of weight reduction with simultaneously improved safety. New TRIP (TRansformation Induced Plasticity) and TWIP (Twining Induced Plasticity) steels containing 15-30 wt. % Mn, amongst other elements, demonstrate excellent elongation properties and high energy absorption capacities.

Different direct reduction processes producing sponge iron use ore fines as raw material. Reduction tests with mixtures of both Mn and Fe ore were carried out in order to represent a pyrometallurgical step (e.g. direct reduction) as a complement to FeMn, aiming to produce a kind of Mn containing sponge iron.

There is no industrial scale direct reduction process for Mn. The present work should make a contribution to the development of a metallurgical process for Mn based on the direct reduction and smelting reduction. For these investigations of reduction of both briquettes and fine ores seven different experimental apparatuses were used, two of them in Brazil and five in Germany.

2 EXPERIMENTAL METHODS

In this work an assessment to kinetic reduction of Mn-ore-charcoal-briquette is presented and it was carried out at the Ironmaking Group of the Department of

Metallurgy and Materials Science (DCMM) at PUC-Rio. The other experiments of both direct and smelting reduction using briquettes and fine ores were carried out at Department of Ferrous Metallurgy (IEHK) at RWTH Aachen University.

2.1 Kinectic Assessment of Manganese Ore Reduction

The briquettes reduction performed at DCMM at PUC-Rio used two electrical tubular furnaces with maximal temperatures of 1200°C and 1400°C, both operating under a controlled atmosphere using a flow rate of 2.5 l/ min N_2. The selected temperatures were 900°C, 1000°C, 1100°C, 1150°C, 1200°C and 1300°C, the tests being performed in a time range from 10 to 90 min. After the reduction stage, every briquette was submitted to optical and electronic microscopy and thoroughly ground for chemical analysis.

2.1.1 Materials: kinetic assessment of reduction of Mn-ore-charcoal briquette

The samples presented in this work are self-reducing briquettes in the shape of cylindrical disks. These briquettes are composed of Mn ore and charcoal as reductant with 20% of stoichiometric excess for carbon. Both of them were finely ground to below 0.15 mm. The green briquettes were pressed under a load of approx. 1 kN and then cold cured.

2.2 Direct Reduction: Briquettes A, B, C and Fine Ores Mixtures

Figure 1 shows the experimental work through direct reduction that has been performed at IEHK.

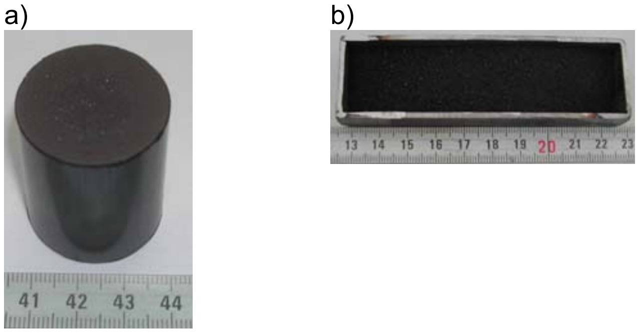


Figure 1: Possibilities to produce Mn containing sponge iron using direct reduction processes with reducing agents based on coal and gas; a) Mn-Fe-ore-coal-briquette and b) sample container with fine ores before reduction

Reduction of various combined mixtures of Mn ore, Fe ore and coal has been investigated in the temperature range from 1000 °C to 1400 °C. The effect of the following parameters as well as the type of reducing agent was investigated: briquette and fine ore mixtures, reduction temperature, reduction time of 10 to 120 min and gas atmospheres, in vol. %: 100% Ar; 10%CO-10%-H_2-80%N_2; 20%H_2-80%Ar; 100% H_2. A flow rate of 2.5 l/min for these gas mixtures was used.

In the reduction experiment with coal, the coal is mixed direct with the ores. In reduction experiments using reducing gases, the coal was in the same way added to the mixture.

2.2.1 Materials: direct reduction of briquettes A, B, C and fine ores mixtures

Tables 1 to 4 show the material used for the experiments at IEHK. More than 15 fine ore mixtures of different compositions have been investigated. In this work the result of only four fine ore mixtures will be presented.

All briquettes manufactured at IEHK and presented in this work have a cylindrical form and measure 30 mm x 40 mm (ϕ x h) (Figure 1). The experimental set-up and procedure are described in [2,3,4,5].

Table 1: Chemical analyses of manganese and iron ores

	Constituents in weight %							
Ores	**Mn**	**Fe**	**MgO**	**SiO_2**	**CaO**	**Al_2O_3**	**P**	**Grain size (mm)**
Mn ore	52.30	6.50	0.10	2.50	0.08	4.95	0.11	0.045…0.18
Fe ore	2.40	67.30	0.08	2.30	0.02	0.40	0.02	0.125…0.125

Table 2: Chemical analyses of used coal

Constituents in weight %	volatiles	Ash	moisture	fixed C	Grain size (mm)
Coal A	2.4	10	12	87.6	0.045…0.125
Coal B	35.4	7.3	23.9	57.2	0.045…0.09
Coal C	20.8	9.1	3.6	70.1	< 0.50

The reduction of briquettes consisting of a blend of Fe ore and Mn ore combined with coal as reducing agent is being investigated within the range of 1000 and 1400 °C. The components of the briquettes were sieved to the required grain size distribution and then mixed with the coal. This blend was then agglomerated in a hydraulic press to the shape of a briquette using a pressure of 1.5 to 2.1 kbar at room temperature, figure 1. The chemical composition is shown in table 3. The chemical compositions of the raw materials are given in Tables 1 and 2.

Table 3: Composition of briquettes

Constituents in weight %	Mn ore	Fe ore	Coal A	Coal C
Briquette A	46.3	34.9	-----	18.8
Briquette B	28.90	51.60	19.50	-----
Briquette C	78.50	-----	21.50	-----

Table 4: Composition of samples in wt. % for fine ores reduction

Sample number	Fe ore	Mn ore	Coal	
			A	B
1, 14	-----	100	-----	-----
3	60.8	39.2	-----	-----
15,	64.1	35.9	-----	-----
17, 24	52.0	28.7	19.3	-----

2.3 Smelting Reduction: Manganese Ore Injection and Manganese-Ore-Aluminum-Briquette Smelting

For better understating of the reduction of Mn oxide through reducing agents like C, Al and Si, the injection of Mn ore into the hot metal and steel melt pool was carried out at IEHK. Reduction temperatures varied from 1500 °C to 1600 °C, reduction time was approx. 60 min and slag basicity ($CaO/SiO_2*Al_2O_3$) depending on the experiment varied from 0.5 to 1.5. The materials used to form the melt pool are hot metal and technical iron (Armcoeisen). The smelting of Mn-ore-Al-briquette was also investigated. Figure 2 shows experimental work through smelting reduction carried out at IEHK.

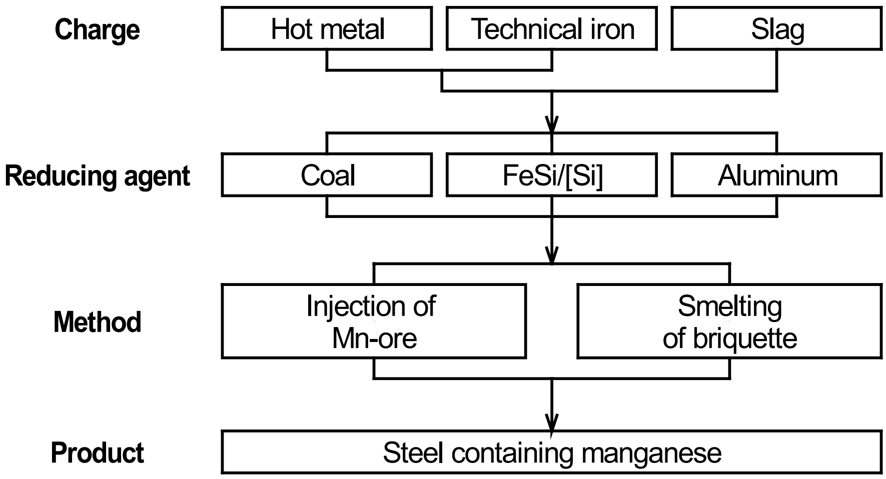


Figure 2: Possibilities to produce steel containing Mn using the smelting reduction processes employing coal, FeSi and Al as reducing agents

2.3.1 Materials: Smelting reduction - Mn ore injection

Tables 5 shows the chemical analyses of the Mn ore used for the injection experiments using Mn ore in the melt pool and the smelting of briquettes, both performed at IEHK.

Table 5: Chemical analyses of Mn ore used for injection and Mn-ore-Al-briquette (wt. %)

Mn	Fe	MgO	SiO_2	CaO	Al_2O_3	P	Grain size in mm for Mn ore injection
46.90	5.50	0.27	3.16	0.17	6.55	0.10	0.8…1.2

Table 6: Composition of Mn-ore-Al briquette and grain size of constituents

Component	Mn ore	Al	CaO
wt. %	69.8	27.7	2.5
Grain size in mm	< 0.2	0.1…0.16	< 0.1

2.3.2 Experimental set-up: Mn ore injection and Mn-ore-Al-briquette smelting

Direct alloying of steel by Mn ore injection is presented as an alternative to replace Mn ferroalloys in part.

The experiments were conducted in a basic-acid lined induction furnace at the IEHK with the use of an injection rig and a lance (see figure 3). 0.5 kg of Mn ore was injected from above the melt surface into 12 kg hot metal with 3.24 wt.% C, 2.12 wt.% Si and 0.22 wt.% Mn via argon with 30 Nm^3/h at the lance exit. The work temperature was 1570-1590°C. The cover slag with the basicity 1.5 was added before the injection to the melt surface. During the experiment the temperature was measured and the samples of melt and slag were taken.

Additionally, the chemistry of the Mn via the injection of Mn ore into the melt pool by various reducing agents such as C, Al and Si and the influence of slag formation on the reduction was investigated. Mn ore with about 47 wt. % Mn_{total} was injected into the melt pool. The theoretical consumption of Mn ore and reductants per ton of crude steel (cs) produced is shown in figure 3 (on the right). The calculations were made with the program *Factsge* corresponding to the experimental conditions.[6,7]

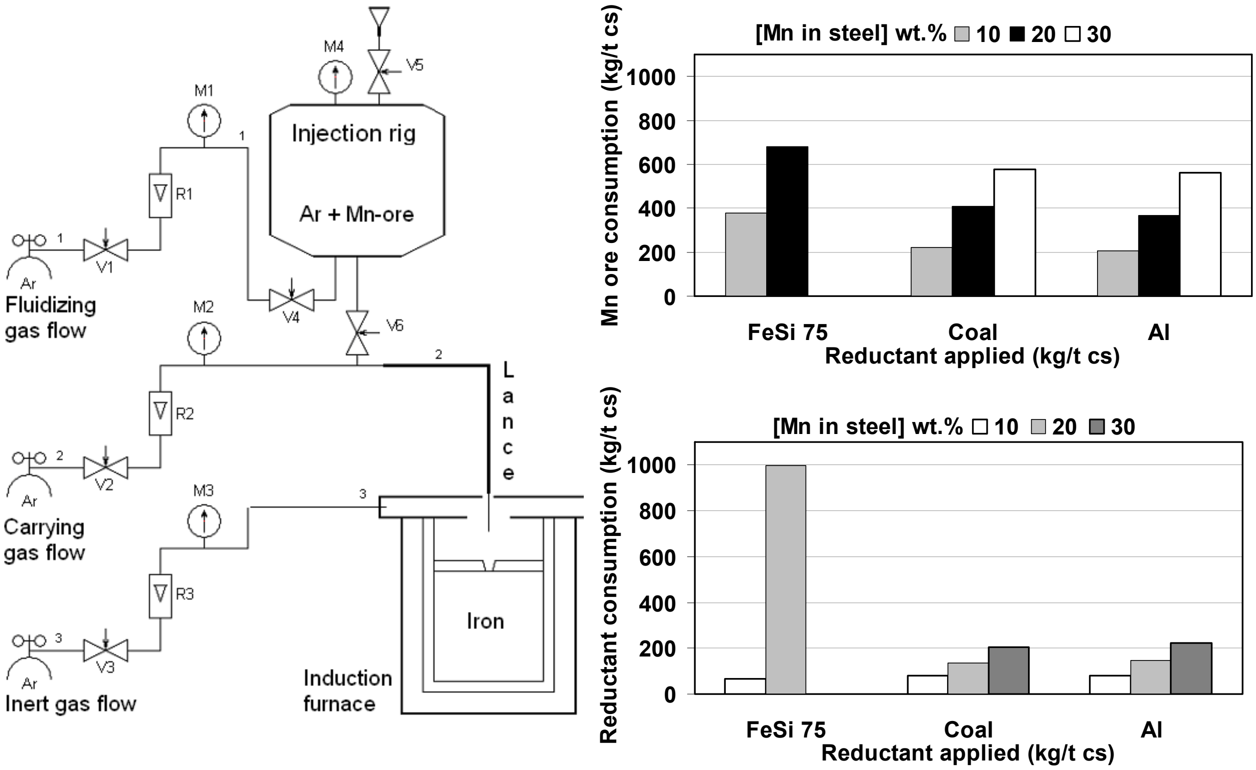


Figure 3: On the left: Experimental set-up for the injection of Mn ore into the melt pool; On the right: Theoretical variation of Mn ore and reductant consumption with reductants FeSi75, Coal and Al for production of steel containing 10 to 30 wt. % Mn

3 EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Kinetic Parameters Evaluation

Based on the experimental results that showed initially crescent conversion curves followed by a further decreasing conversion, a two-stage process mechanism was proposed. The first stage, valid for short reaction times, up to circa 10 min, essentially involving the reduction of MnO_2 to MnO, was followed by a second step, characterized by the transformation of the generated MnO, probably to Mn_3C (Figures 4 and 5).

Aiming at the description of the process as a whole, that is the formation of the MnO for the first stage and a second stage where the transformation of the previously generated MnO takes place, the equations 1 and 2 were utilized. The first parcel on the right side of equation 1 models the MnO formation and the second its consumption. The values for k_o (pre-exponential frequency factor) and E_a (apparent activation energy) are also presented for both process steps.

$$\chi_{MnO} = \chi_1 [1 - e^{-t/\tau 1}] - \chi_2 [1 - e^{-t/\tau 2}] \qquad (1)$$

The Arrhenius equation (2) was utilized and ln (χ_i / τ) was considered as a function of the temperature reciprocal (1/T). The graphs in Figures 6 and 7 present curves that characterize the mechanisms occurring in the reduction process for short reaction times and for the second stage.

$$\ln (\chi_i / \tau) = \ln k + [- E_a / RT] \qquad (2)$$

3.1.1 Temperature effect

Figures 4 and 5 present the kinetic curves for the experimental and conversion degrees for the first and second stages of the model.[8]

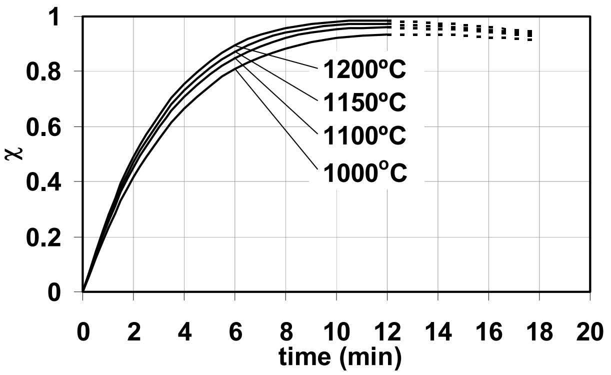


Figure 4: MnO_2 to MnO conversion changing with reduction time at different temperatures of reduction

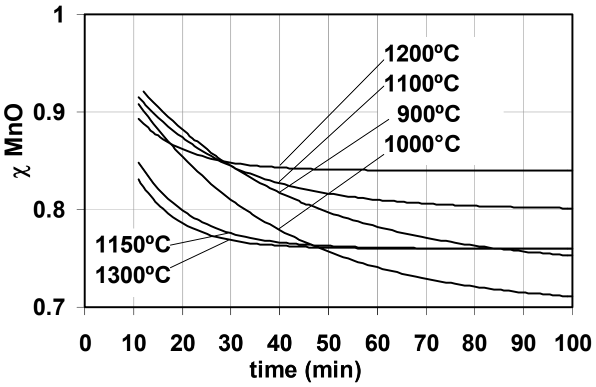


Figure 5: Decrease of MnO_2 to MnO conversion, due to Mn_3C formation, changing with reduction time at different temperatures of reduction

Figure 4 shows that the self-reduction of the Mn-ore-charcoal-briquette exhibited high conversions for Mn^{+4} towards Mn^{+2}, being increased for higher temperatures, mainly for those above the reductant reactivity (800 °C). It was also determined that the adopted kinetic model described with accuracy the reduction of briquette (correlation of 94.4 % for the short times step and 96.1 % for the second step). The analysis of Figures 4 and 5 permits the conclusion that for temperatures above 900 °C the MnO_2 to MnO conversions are high.

For the first stage (MnO_2 to MnO), the calculation shows $E_a = 11.5$ kJ/mol and $k_o = 57\text{x}10^{-6}$ s^{-1}; these values suggest a diffusion control. Second stage (MnO to Mn_3C) shows $E_a = 46.1$ kJ/mol and $k_o = 48\text{x}10^{-6}$ s^{-1}; these values are consistent with a mix control (Figure 6 and 7).

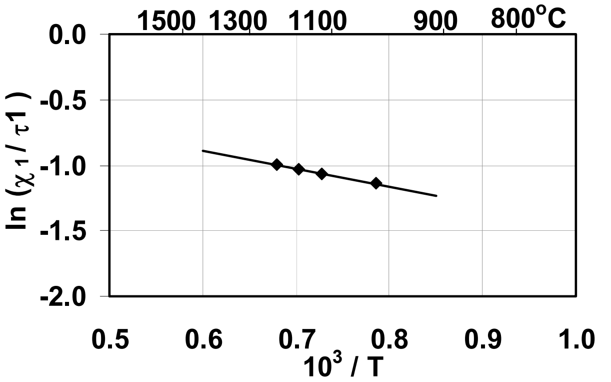


Figure 6: Arrhenius plot for the first stage of reduction (MnO_2 to MnO) from 1000 to 1200 °C

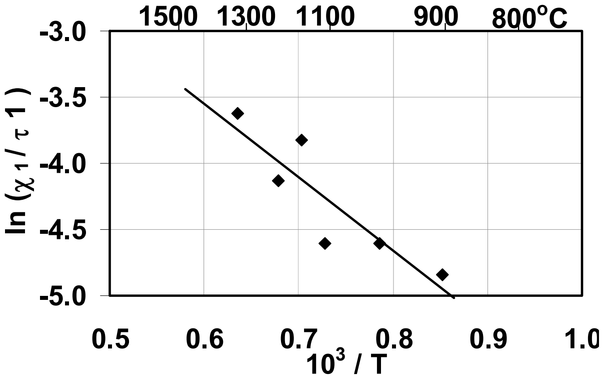


Figure 7: Arrhenius plot for the second stage of reduction (MnO_2 to MnO to Mn_3C) from 900 to 1300 °C

3.2 Direct Reduction: Briquettes A, B, C and Fine Ores Mixtures

3.2.1 Reduction of Fe-Mn-ore bearing briquettes

In order to evaluate the addition of Fe_2O_3 as Fe ore in the reduction rate, trials were accomplished with briquettes A, B and by 1200 °C. The degree of metallization (MD) is calculated by equation (3). X is obtained from the chemical analyses with the contents (wt. %) of Fe and Mn.

$$\text{Metallization degree} - \text{MD}\,(\%) = \frac{X_{\text{metallic}}}{X_{\text{total}}} \times 100 \tag{3}$$

Figures 8 and 9 show the briquettes before, during and after reduction experiments.

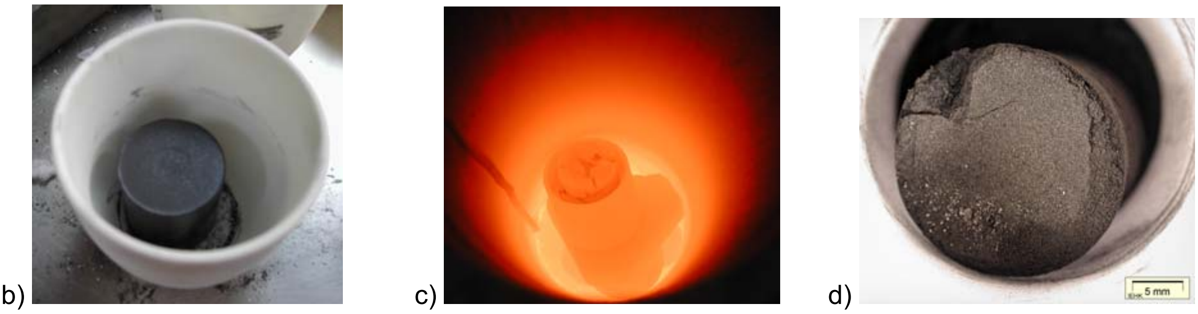


Figure 8: Briquette in the crucible: a) before reduction, c) during reduction, d) after reduction at 1000° C and 50 min reduction time

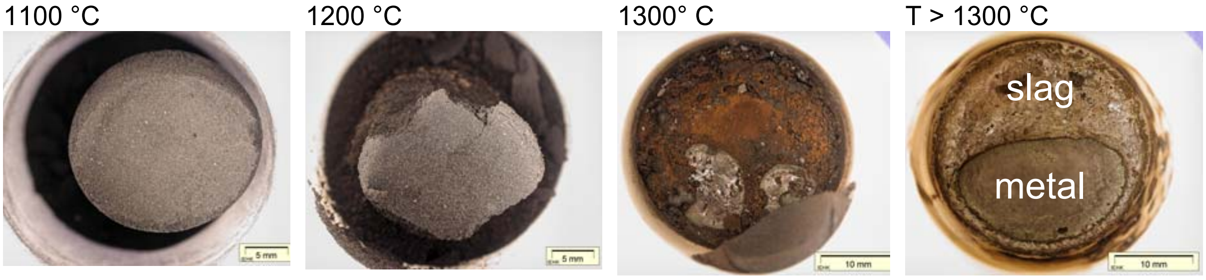


Figure 9: Briquette A after reduction from 1100 to 1300 °C, 50 min of time of reduction and briquette B at T > 1300° C, molten phase of slag and metal

Figure 10 on the left shows the added and the stoichiometric carbon content of briquettes A, B and C before the reduction. The reduction rate of Mn increases markedly by adding Fe ore in the ore mixture (briquette B, Figure 10 on the right). [5]

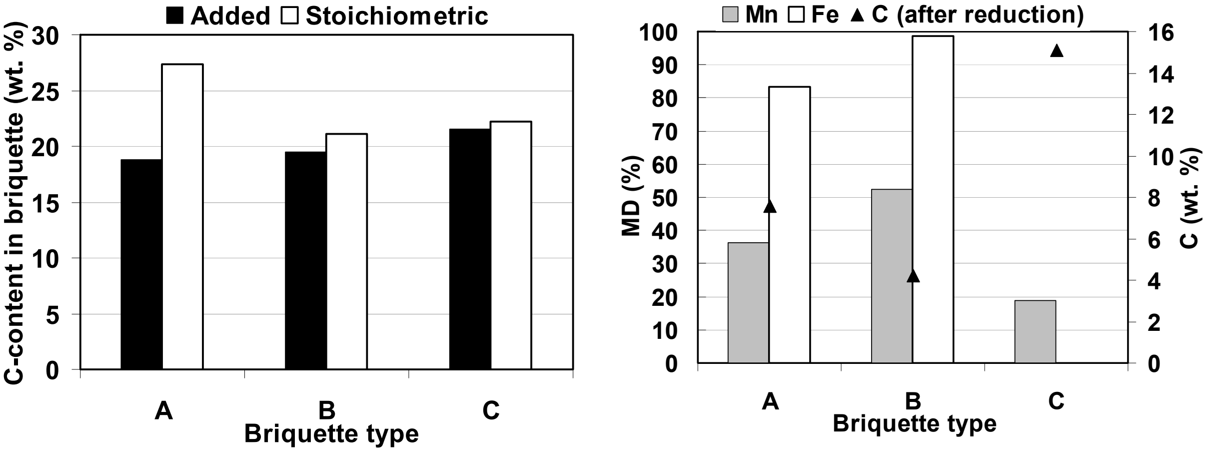


Figure 10: On the left: Comparison of both stoichiometric and added carbon content of briquettes (A,B,C); On the right: Metallization degree (MD) for Fe and Mn of trials at 1200 °C, 50 min of reduction time and 2.5 l/min Ar as function of briquette composition and C-content after reduction

Reduction of briquettes above 1300 °C and for more than 30 min of reduction time results in a molten phase. This liquid phase is divided as slag and metal. The metal part has been chemically analyzed. Figure 11 shows the reduced briquettes B after reduction and presents the contents of P, Si, C and Mn in wt. % in the metal part against the temperature and time of reduction. The rest of the metal part consists mainly of Fe. The content of C is about 3.5 wt. % and is neither affected by the reduction temperature nor by the duration time. A reduction temperature of 1400 °C and a duration time of 30 min led to a metallic phase that is practical for a commercial use. The inner diameter of the crucible in Figure 11 is 35 mm.

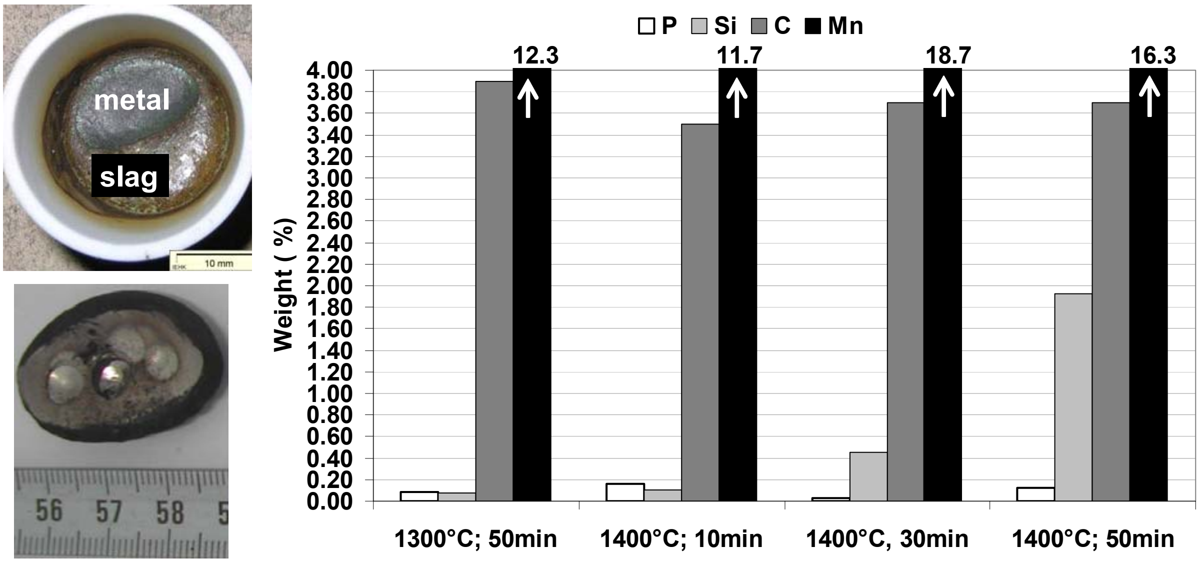


Figure 11: On the left: Briquette B after reduction at higher temperatures up to 1300 °C (above); Metal part with small drilling cavity (below). It was drilled in order to take sample for chemical analyses; On the right: Contents of P, Si, C and Mn in the metal part after reduction for various temperatures and reduction time under 100 % vol. Argon, rest mainly Fe

3.2.2 Reduction of fine ores mixtures

The suitability of reduction was analyzed using an electron probe micro analyzer (EPMA) and X-ray diffraction (XRD) examinations (Table 7 and Figure 12).

The results of mixture consisting of Mn and iron ores and coal (sample 17) show the following:[5]

- No carbide phase was detected with XRD at 1000 °C. The identified phase consists mainly of Fe with slight presence of mix crystal phase as (Fe,Mn)O, SiO_2 und 2(MnO)*SiO_2.
- Micro analyzer analysis shows metallic Fe phase with low amount of Mn. The absence of oxygen in the reduction product confirms that Fe and Mn are present in metallic form.
- The XRD measurement identifies only Fe and no Mn, in samples in which the chemical analysis shows Mn. This is an indication that Mn is present dissolved in Fe because the Fe mix crystal with Mn amount is detected on XDR as Fe.
- A Fe-Mn mix crystal phase with Fe as main phase is identified as Fe by XRD. This is because the small amount of Mn only changes the crystal lattice slightly.

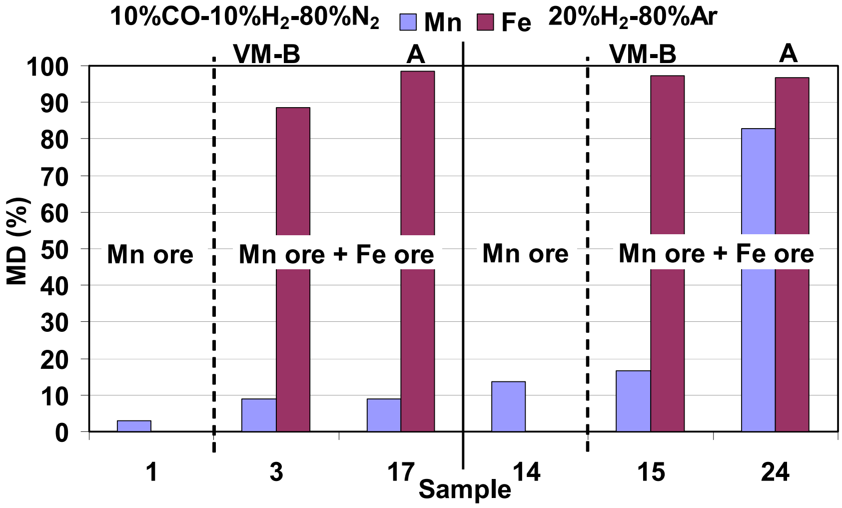
Table 7: Results of examination of samples 1 and 17; X-ray, chemical and analysis with EPMA

Method	**X-ray**	**Phase analysis with EPMA (electron probe micro analyzer)**	**Chemical analysis**
sample	phase	wt% at point of analysis	wt. %
1	Fe MnO 2(MnO)*SiO_2 FeO 2(FeO)*SiO_2 MnO_2	**1-7:** 0.25% Mn, 99.17% Fe **8-11:** 23.4% O, 71.7% Mn, 3.8% Fe **16-20:** 0.7% O, 0.51% Mn, 98.2% Fe **27-29:** 35% O, 1.4% Mn, 20% Fe, 26% Si **30-32:** 23% O, 6.6% Mn, 69.3% Fe **33-36:** 31.7% O, 4,2% Mn, 50% Fe, 13% Si	48.3% Fe_{met} 1.5% Mn_{met}
17	Fe MnO SiO_2	**1-3:** 99.9% Fe **8-10:** 53.7% O, 45.2% Si **11-18:** 96.6% Fe, 3.3% Mn **46-49:** 95.3% Fe, 4.5% Mn	56.1% Fe_{met} 1.5% Mn_{met}

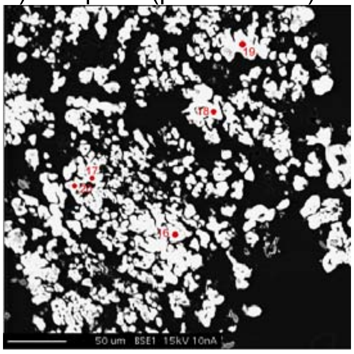
An increased MD is achieved by adding Fe ore to Mn ore. It suggests that the presence of metallic iron reduces the activity of metallic manganese, because it is absorbed by the metallic iron by diffusion. The diffusion of Mn in Fe results in Mn lower activity, because Mn is absorbed by Fe and thus is no more in direct contact with the reduction reaction. New Mn must be formed so that the equilibrium between Mn oxide and Mn is again established:

$$MnO + C \Rightarrow [Mn] + CO$$

In this way more Mn is reduced than in the reduction without the presence of Fe.
The MD for both Mn and Fe rises with increasing content of H_2 in the gas mixture. The use of coal A and of volatile matters of coal B (VM-B) through the reduction with 20%H_2-80%Ar raises the MD for Mn and Fe. The reduction is inhibited by the gas mixture of 10%CO-10%H_2-80%N_2 (Figure 12). [5]



a) sample 1 (points 17-20)



b) sample 1 (points 40-45)

c) sample 17 (points 1-10)

d) sample 17 (points 11-33)

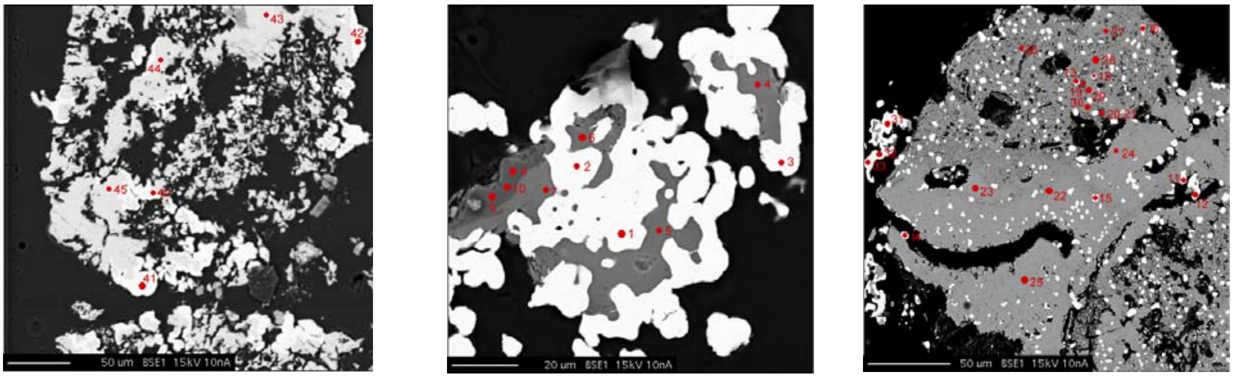


Figure 12: Metallization degree of samples at 1000 °C reduction temperature and 120 min reduction time, 2.5 l/min of 20%H_2-80%Ar (in vol. %) as reduction gas, in muffle furnace 1; From a) to d) microstructure of samples 1 and 17 showing points analyzed using EPMA

3.3 Smelting Reduction: Manganese Ore Injection and Manganese-Ore-Aluminum-Briquette Smelting

3.3.1 Experimental and modelling results of Mn ore injection into the melt pool

Results of injection experiments carried out at IEHK were compared with the previous work of Vargas-Ramires et al.,[7,9] where the reduction of MnO from the molten slag with liquid steel of high carbon content was investigated. Figure 13 shows a higher rate of reduction through the injection of Mn ore into the melt. It can be explained by the greater reaction surface. The aim of modelling is to estimate the depth of penetration and the residence time in the melt for injected Mn ore particles.
Figure 14 shows the results of Mn-yield of the injection of Mn ore into the melt pool. The reducing agents like C or Si from the hot metal and the addition of Al showed better results than FeSi75.

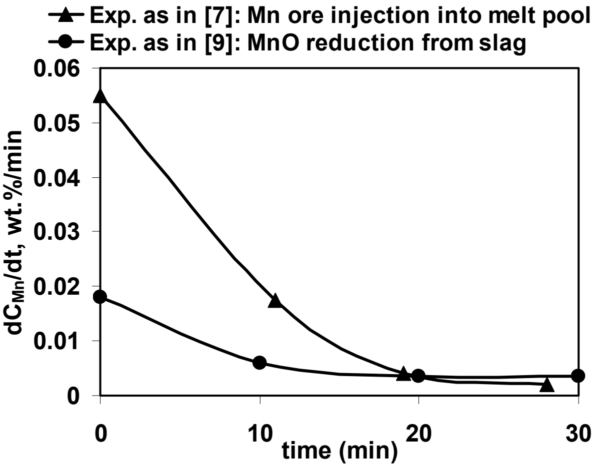


Figure 13: Rate of Mn reduction, comparison between injection and reduction from slag at 1600 °C

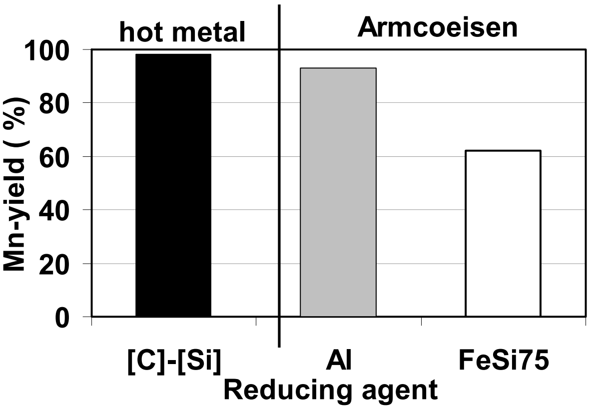
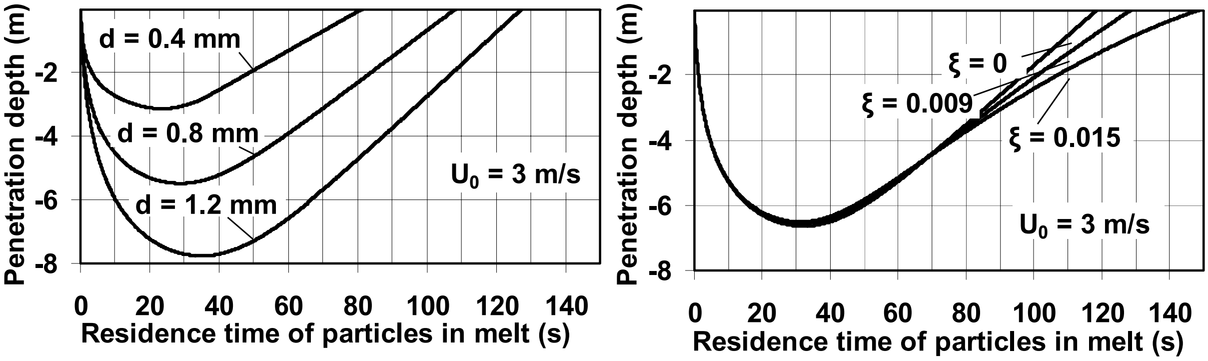


Figure 14: Mn yield of smelting reduction experiments at 1600 °C, 60 min duration time. Reducing agents: C and Si from hot metal and for Armcoeisen Al and FeSi75 were added

Results of modelling calculations are shown in Figure 15. The rate of reduction is higher with the injection of Mn ore into the melt than with reduction from the slag. The longer residence time in the melt corresponds to the bigger particle, due to deeper penetration. The longest residence time of the particle in the melt is less than 2.5 min.

The modelling results shown in figure 15 can be taken as the upper limit of residence time of particles in the melt. Currently the model is in further development with regard to the kinetics of reduction and dissolution of Mn ore particles.[10]



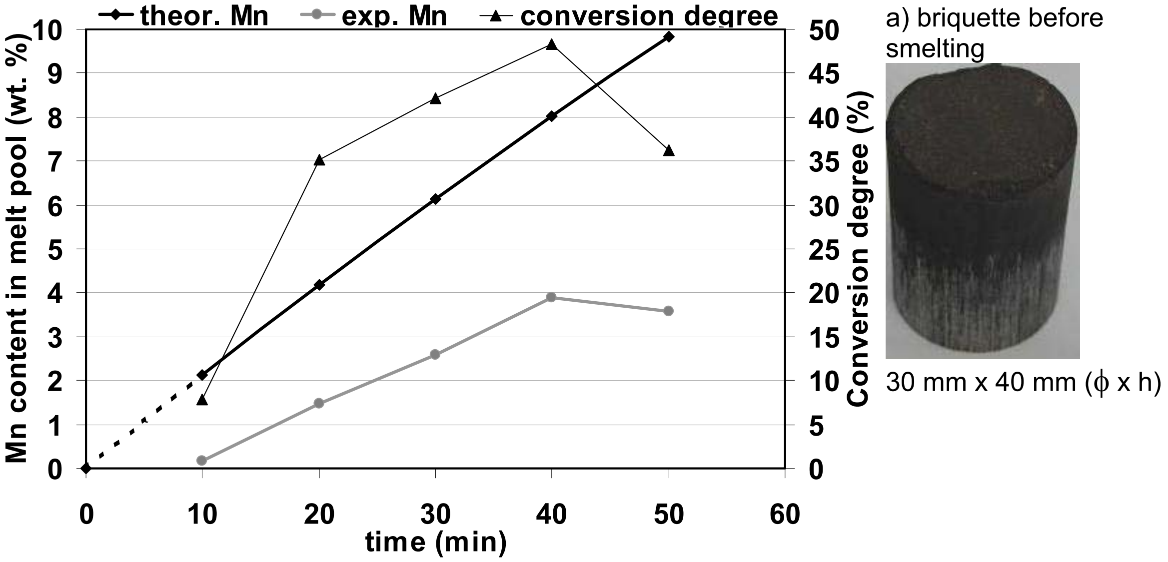
a) particle is not dissolving in the melt

b) particle is dissolving in the melt

Figure 15: Modelling results of Mn ore injection from above the melt surface

3.3.2 Experimental results of manganese ore aluminium briquette smelting

Figure 16 shows the result of feeding Mn-ore-Al-briquettes into the melt. It shows an average conversion degree of approx. 40 %. Using this alloying concept an enrichment of 4 wt. % of Mn of the melt can be reached. However, the slag formation and the Mn losses into the slag are still a challenge and the subject of further investigations. In addition, the price of aluminum has to be considered for an economical process.[5]



a) briquette before smelting

30 mm x 40 mm (ϕ x h)

Figure 16: Mn Content in the melt pool as function of time at app. 1600 °C, theoretical and experimental Mn content, conversion degree, smelting reduction with Mn-ore-Al-Briquettes; a) Mn-ore-Al-briquette

4 CONCLUSIONS

The proposed kinetic model for the reduction of Mn-ore-charcoal briquette is of a semi-empiric nature and matched consistently the experimental results.

An increased MD is achieved by adding Fe ore to Mn ore. It suggests that the presence of metallic iron reduces the activity of metallic manganese, because the metallic Mn is absorbed by the metallic iron by diffusion.

Results have shown that metallization degrees for Mn of more than 70 % can be reached using combined direct reduction of Fe ore and Mn ore. The use of carbon as reducing agent (CO, coal), however, leads to carbon contents in the products that are too high for some applications. The direct reduction via H_2 as reducing gas can control the carbon content. Phosphor content in Mn ore must be very low.

Injection of Mn ore into the melt is an alternative to steel alloying. However, this method can be applied only for the production of alloys with a maximal Mn content of 3 wt. %. The developed model can be taken as the upper limit of residence time of particles injected into the melt.

The accomplished experiments presented the possibility of adding Mn by direct and smelting reduction. This work already gives ideas for further developments for industrial scale applications.

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