



From Oxide Residues of Al-slag-treatment to SiAl-masteralloys via carbothermic reduction

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Abstract

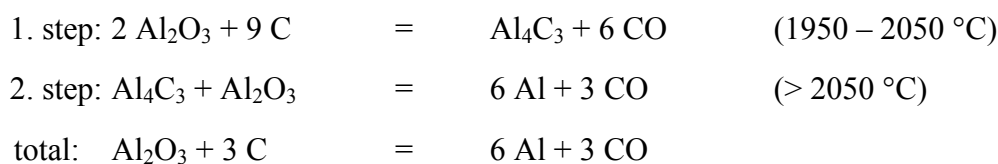
At present there no sustainable concept exists to process the non metallic, mostly oxidic residues emerging from the treatment of salt slags of the secondary aluminium production. The current state of the art in Europe is still landfilling or a partial use in the cement industry. These alternatives both involve financial expenses combined with the loss of the aluminium content. Until now, no feasible solutions are proven in scientific research dedicated to the recycling of these materials. Due to the fact that 50 % of the worlds aluminium production is used for AlSi-based cast alloys in the automotive industry, the proposed process targets on the joint carbothermic reduction of oxidic aluminium and silicon residues to produce AlSi master-alloys. AlSi alloys are traditionally produced by adding elementary silicon to the aluminium melt. Whereas the conventional production of Aluminium requires a high amount of energy, a high purity of the raw material and is related with high emissions, the development of a process for reduction of secondary raw materials could reduce the expenditure of energy and minimises the use of primary raw material improving the environmental sustainability. Additionally, the possibility to recycle the predominantly oxidic residues of secondary aluminium production, allows for further reduction in resource demand like landfilling space or transport issues. This paper presents first results of a four years research project at IME Aachen and will show, that it is possible to produce an AlSi alloy in an electric arc furnace by carbothermic reduction of Al_2O_3 and SiO_2 .

1. Background and Fundamentals

Numerous investigations were carried out to develop carbothermic reduction of aluminium oxide but up to now no metallurgical process was accomplished to produce metallic aluminium. The reactions for the reduction of Al_2O_3 and SiO_2 to produce Al or an AlSi alloy are very complex. Numerous components (carbides, oxycarbides, oxides, suboxides) are formed and react during the process. Therefore various theories about the reduction process exist and has to be considered critically because the process is not completely investigated and well known. For this reason only a short overview can be given of the process in the following.



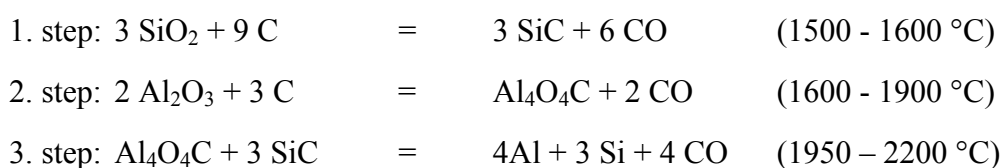
In [1] and [2] it was shown, that the required reduction temperature of aluminium oxide varies between 2 100 and 2 300 °C and the process runs in two process steps:



Due to the high temperature the metallic aluminium generated has a high vapour pressure resulting in a high amount of gaseous Al and Al suboxides. Therefore the prevention or at least the minimisation of aluminium losses is a big challenge for a carbothermic process. The main problem of the process is the solubility of aluminium carbides and oxides. They are formed during the process, dissolve in Al and must be separated, which is virtually impossible [3]. Research was carried out by Pechiney (1955 - 1967), Reynolds (1971 - 1984) and today especially by Alcoa / Elkem who base on the research of Reynolds. Here the reaction takes place in a split reactor. In the first sector the aforementioned first step reaction takes place and an Al_2O_3 - Al_4C_3 - slag is created. This slag flows into the second sector, where the reduction step happens. The formed metallic aluminium is cleaned from carbides in a subsequent step. Gases which contain aluminium are caught and fed again to the process as dust in order to minimise the losses. The energy input is achieved electrically via horizontal or vertical electrodes [1], [2].

The first carbothermic reduction of aluminium ores for the production of AlSi alloys was carried out by Lurgi-Thermie in Germany from 1925 until the beginning of the 2nd world war and then was abandoned. In spite of continuing research work in the United States the process could not gain commercial acceptance except for Zaporoschje, Ukraine. The advantage of this process is the prevention of the aforementioned solubility of aluminium carbides and oxides in Al. This can be prevented by adding oxides (for example SiO_2), which causes a decomposition of the carbides [3]. The content of aluminium in the emerging AlSi alloy should not exceed 60 % in order to avoid significant carbide formation [4].

The reactions of the process run in three steps:



The process is conducted in an electric arc furnace (less frequently in shaft furnace) at a temperature of roughly 2000 °C. Depending on the required alloy pre-treated materials (pure quartz, kaolin, pure clay, reducing agent) or natural (red/white bauxite, clay, granite, reducing agent) feed material is used. The burden is milled, mixed, briquetted and then charged. The Si-content of the alloy which was produced in Germany was between 40 and 60 %, in Ukraine between 30 and 35 %. If a high quality of the alloy is desired a separation of the slag via salt-cryolite-addition is conducted, alternatively the alloy is cooled down to segregate impurities. In order to reach an amount between 10 and 13 % Si the alloy can be diluted with pure aluminium or the silicon is segregated [4], [5].

The feasibility of the simultaneous carbothermic reduction of aluminium and silicon oxides originating from primary resources is already proven. The basis is the decomposition of Al carbides due to the presence of SiO₂. The fundamentals were already researched in the Soviet Union but were published only to a very small extent. The transfer of this process concept to secondary resources, for example originated from secondary aluminium industry, hasn't been investigated yet. But the marginal published results can be used as basis for further researches in order to investigate, adapt and optimize this process, which is currently the topic of a PhD project in Aachen.

2. Aim of the research

The aim of the project is to show the general feasibility of a carbothermic reduction process for the NMR (non metallic residues from Al-salt slag treatment) of this material in order to produce an AlSi alloy. Table 1 shows the planned process.

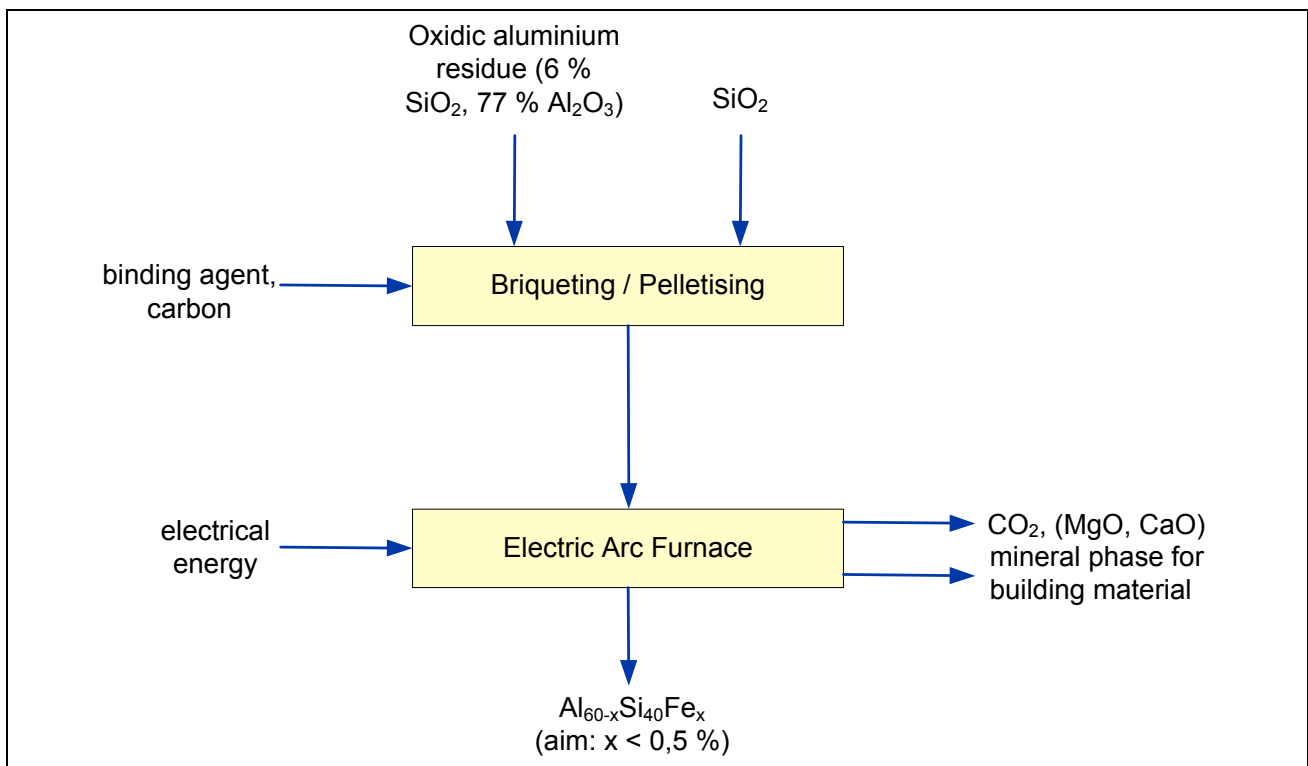


Figure 1: General view of the planned process

The following points have to be investigated theoretically as well as by experiments:

- Transfer and adapting of the process window of the primary raw material towards secondary materials
- Selection of suitable silicon feeds to the residues of the secondary aluminium industry (quartz or sand, other secondaries)
- Suitable grain size of the SiO₂-carrier



- Amount, size and type of the reduction agent
- Behaviour of the alkali metals and Halides present in the residues
- Type and amount of binder for briquetting or pelletising
- Necessary pressure for briqueting in order to provide the required stability
- Process sequence (temperature, charging, height of pellet bed) of the reduction of the briquets / pellets in the electric arc furnace
- Treatment of the resulting Al-30 % Si raw alloy to a commercial master alloy with 10 – 13 % Si (market requirement on the alloy, purity)
- Possibility of recycling of dusts/slugs (Al- and Si-carbides) in the process in order to design a waste free process.
- Material efficiency, energy balance and environmental effects, economic efficiency

The research is split in two parts. The first part is the thermochemical calculation of the behaviour of the material in the furnace depending on the SiO_2 amount and the temperature. The calculations are conducted with the software FactSage[®] using the databases SGTE, FTOxid, FSlite and FSupsil. The second part is the practical implementation of the results in the electric arc furnace.

3. Thermochemical Calculations

The aim of the thermochemical calculations is to investigate the influence of SiO_2 on the reduction process. As described in chapter 1 the winning of aluminium by a direct carbothermic reaction is very difficult unless impossible. The addition of SiO_2 can reduce the formation of carbides. The higher the amount of silicon in the alloy the lower is the formation of carbides. However the silicon amount in the alloy should be as low as possible considering of the later use of the alloy.

The calculations consider the distribution of aluminium in the different phases between 1 500 °C and 2 400 °C. The amount of carbon is stoichiometric relative to the total oxide content in the material. The amount of the NMR is kept constant, the amount of added SiO_2 is related to the targeted amount of silicon in the emerging alloy. The distribution of aluminium in the phases is calculated for an alloy with an targeted amount between 10 and 70 % silicon. The following diagrams show exemplary the distribution of an Al alloy with 30 and 50 % Si. By reasons of clearness, phases which only appears in a amounts are not considered in the following results.

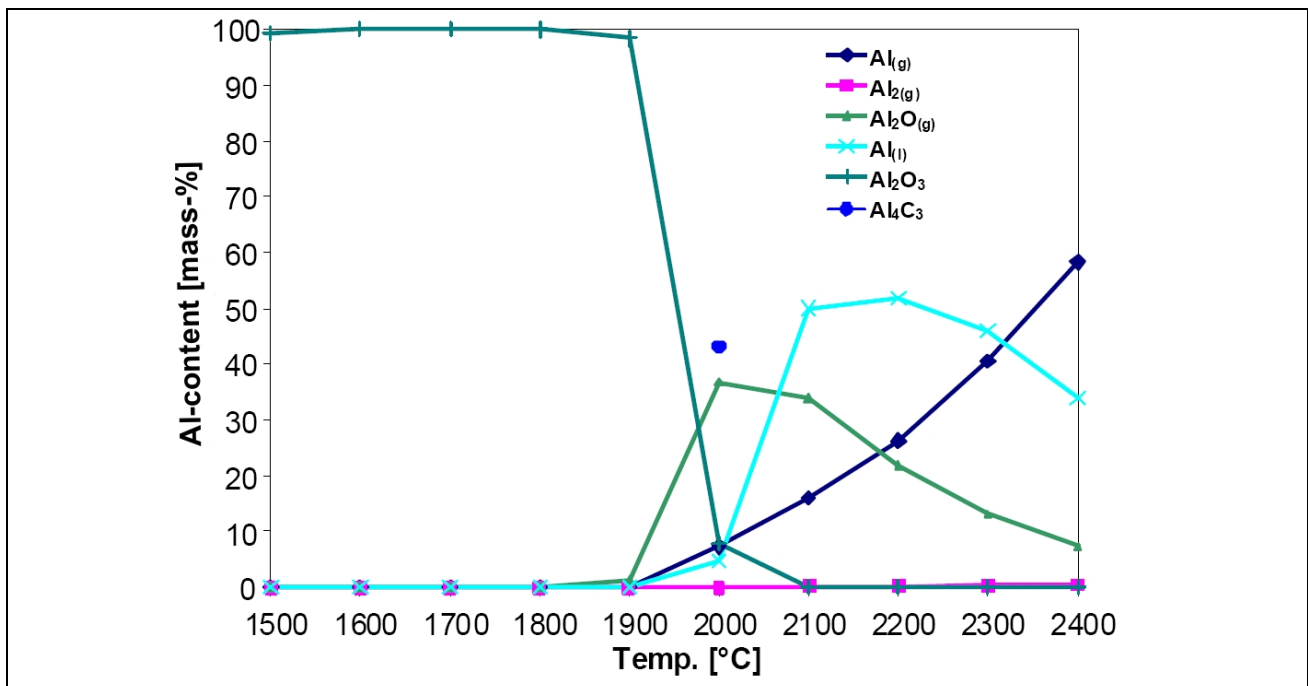


Figure 2: Phase distribution of aluminium at the production of an AlSi30 alloy

Figure 2 shows the distribution of aluminium at the production of an AlSi30 alloy between 1 500 °C and 2 400 °C. The reduction of Al_2O_3 starts slowly at 1 800 °C and ascend steeply at 1 900 °C. The gaseous phase increases continuously and contains gaseous Al and Al_2O . The composition of this phase varies and the amount of gaseous Al increases with increasing temperature. The maximum of Al_2O is reached at 2 000 °C (~38 %) and the highest amount of liquid aluminium is reached at approximately 2 200 °C (51 %). At 2 000 °C the only carbide phase Al_4C_3 appears.

In figure 3 the distribution of aluminium is shown at the production of an AlSi50 alloy between 1 500 °C and 2 400 °C. The distribution is similar to figure 2. The formation of metallic aluminium starts at approximately 1 900 °C. The liquid Al-phase reaches again the maximum at 2 200 °C as well as at AlSi30, but here the maximum is lower and reaches only 32 %. At this temperature the total amount of gaseous aluminium phases (Al(g) , Al_2O) is 67 %. The amount of Al_2O increases strongly up to 2 000 °C and declines at higher temperatures. The quantity of gaseous aluminium rises continuously up to almost 80 % (2 400 °C) and the only carbide phase appears at 2 000 °C.

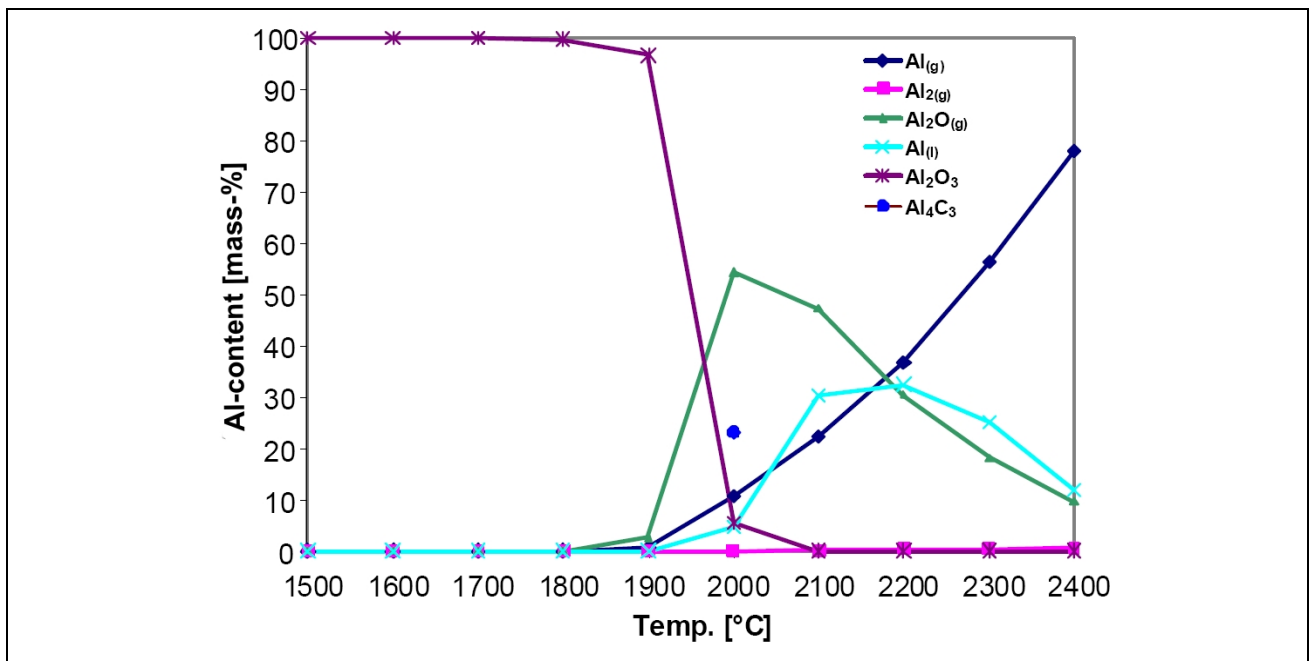


Figure 3: Phase distribution of Aluminium at the production of an AlSi50 alloy

After consideration of both diagrams the maximum of liquid aluminium is at 2 200 °C. Metal phases don't exist under 1 900 °C and the gaseous aluminium phase increases strongly at ascending temperature. Apparently the gaseous Al phase isn't formed by reduction of Al₂O₃ but originates of liquid aluminium because Al₂O₃ doesn't already exist anymore at 2 100 °C. The amount of the carbide phase decreases with increasing silicon amount but the presenting of this phase must be considered critically due to the forming at only one temperature.

Figure 4 illustrates the distribution of metallic aluminium in the gas and liquid phase at a targeted silicon amount between 10 and 70 % at 2 200 °C. The distribution shows that an increasing amount of silicon causes an higher amount of Al containing gases (Al, Al₂O). The gases are lost in the process and so according to this calculation the amount of silicon should be low.

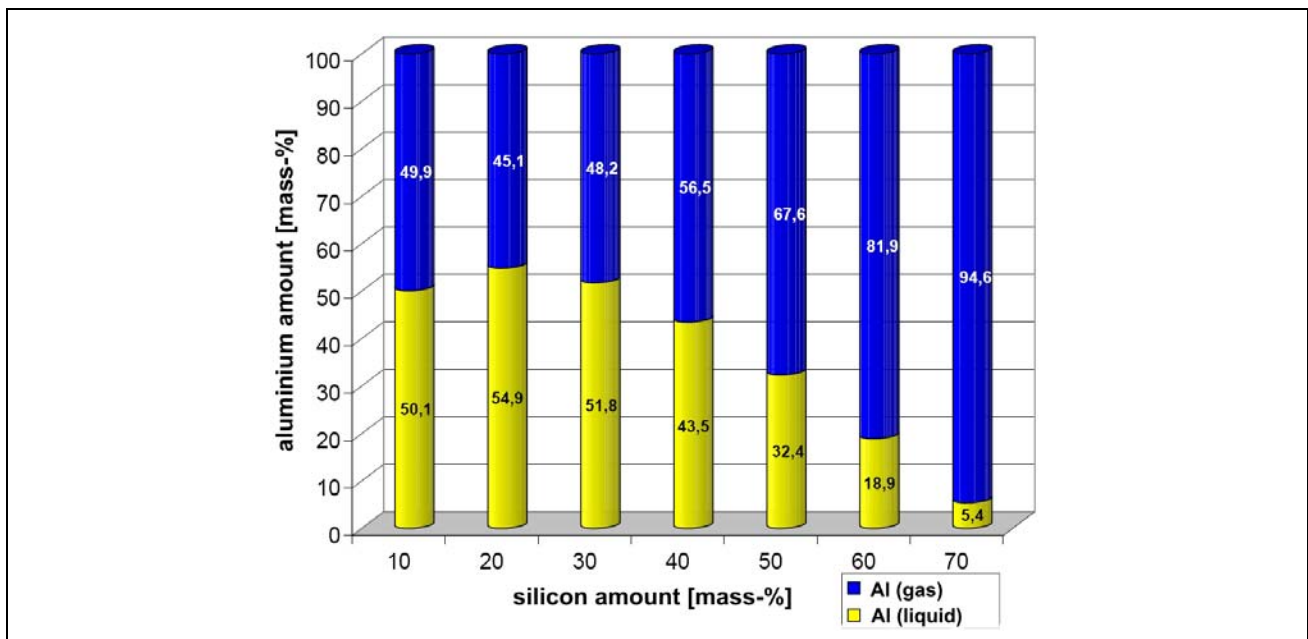


Figure 4: Overview of the distribution of aluminium in gas (Al, Al₂O) and liquid phase at 2 200 °C

Additionally to the investigation about the influence of the silicon, the influence of carbon amount is calculated. The left diagram in figure 5 shows the emerged amount of Al(g), Al₂O (g) and Al (l) subject to the stoichiometrical amount of carbon and the right diagram illustrates the distribution of the carbon to C, SiC and CO phase subject to the stoichiometrical amount of carbon. The calculations are carried out for an 0.5, 0.75, 1 and 1.25 stoichiometrical addition of carbon. Every axis describes a stoichiometrical amount of carbon and the point of intersection of the axis and the line of the substance shows the amount of the substance in mass-%.

The diagrams demonstrate that an 0.5 stoichiometrical addition of carbon isn't sufficient for a complete reduction of Al₂O₃. On the one hand elemental carbon isn't in the system anymore and on the other hand an high amount of Al₂O (50 %) exists because not enough carbon is provided for the reduction. The maximum yield of aluminium is reached with 0.75 stoichiometrical amount of carbon. Higher amounts of carbon cause the same yield of aluminium but the amount of elementary carbon increases from 20 % (0.75) to 40 % (1) and 56 % (1.25). Furthermore the formation of SiC occurs.

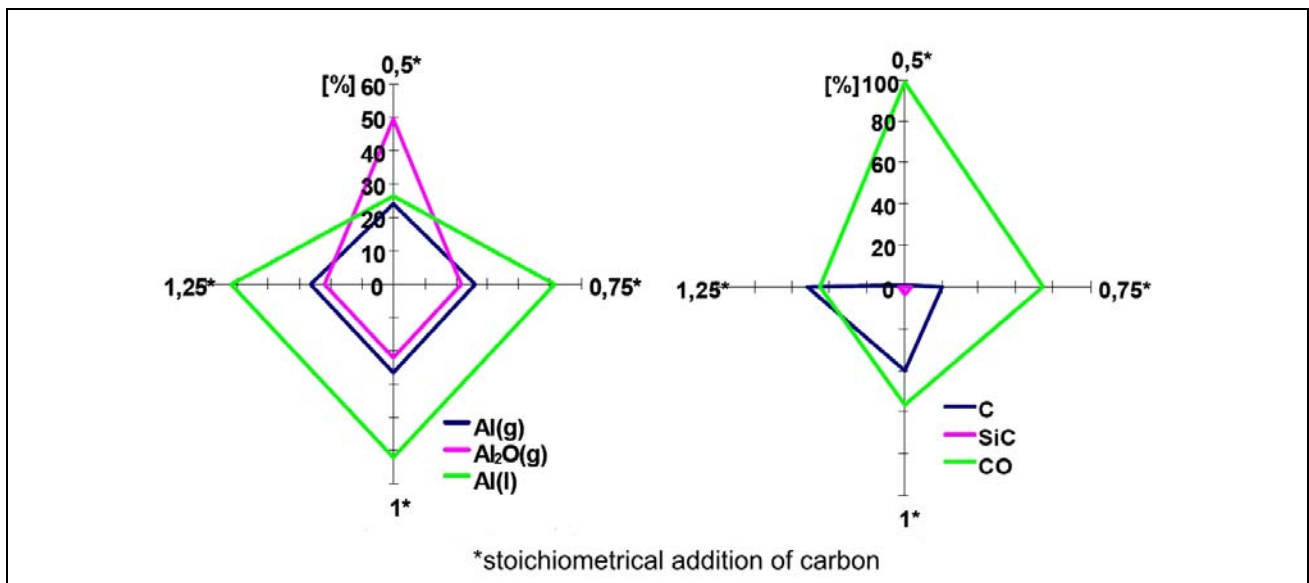


Figure 5: Effect of the carbon addition to the yield of aluminium

The results of the thermochemical calculations can be summarised as follows:

- Highest yield of aluminium should be achieved in the liquid metal phase at 2 200 °C, independent of the silicon amount
- Significant losses of aluminium due to the high process temperature and the associated vapour pressure of Al and Al₂O
- The amount of added silicon directly influences the yield of aluminium. A high amount of silicon in the alloy causes a high reduction efficiency of aluminium but an increase of gaseous aluminium as well. The ideal silicon amount is between 20 and 30 %.
- The highest yield of aluminium is reached by substoichiometric addition of carbon (0.75) relative to the total oxide content in the material. Losses by an early combustion or dust losses are not considered in the calculation

4 Experiment

4.1 Feeding material

The treated NMR originates from Alustockach GmbH (figure 6) and is a product after treatment of salt-slugs by leaching and crystallisation [6]. Table 1 illustrates the composition of the material. The results of the XRD analysis weren't available yet.



Figure 6: Non metallic residue of Alustockach GmbH

Table 1: Composition of the feed material (NMR)

Compound	Al ₂ O ₃	SiO ₂	MgO	CaO	TiO ₂	Na ₂ O	K ₂ O	Fe ₂ O ₃
Amount in %	76 - 78	5 - 8	4 - 6	1.5 - 2.5	0.7 - 1.5	0.2 - 0.6	0.1 - 0.5	0.7 - 1.5

Compound	Chlorides	Fluorides	Al (metallic)
Amount in %	max. 0.5	max. 0.7	0.5 - 2

Due to the fact that the upper grain size of the material is 500 μm and almost 50 % of the material is smaller than 100 μm as well as due the low density ($\rho = 2.95 \text{ g/cm}^3$) an extensive loss by dust carry over during the reduction process can be expected. Because of this grain size the material has to be agglomerated before charging into the furnace. Furthermore a better gas flow and reactivity is provided through the pellet bed in the furnace. Silicon dioxide in the form of quartz sand is added to the aluminium residue in order to adjust the lack of silicon in the material. The quartz sand averages 98 % silicon dioxide. The reduction agent is lignite coke which contains 86 % carbon and is agglomerated together with silicon dioxide and the aluminium residues. Before pelletising, the carbon is ground in a swing mill because first tests show a low agglomeration ability of the coarser carbon particles, which have an average diameter between 0.1 and 1.5 mm. The agglomeration tests are carried out with molasses and sodium silicate and the pellets are dried at ambient temperature and at 150 °C for 24 and 48 h. The pellets which are formed with molasses and which are dried for 48 h at 150 °C show the highest stability, so that these pellets are used for the trials in the electric arc furnace. The pellets have a diameter of 10 and 15 mm and an average resistance of 980 g. The resistance is measured by loading the pellets with weight loads until they break.

4.2 Equipment

The trials are conducted in the AC-laboratory electric arc furnace (Figure 7). The max. power of the furnace is 100 kW and the total volume of the graphite crucible is 6 l. The bottom electrode is a water cooled copper electrode and the top electrode consists of graphite. The position of the top electrode can be infinitely hydraulically adjusted. The diameter of both electrodes is 50 mm. The furnace can be tapped or tilted. The crucible is mounted into the furnace on a coke bed in order to assure a sufficient electrical conductivity between crucible and bottom electrode. The advantage of the graphite crucible is a higher thermal stability as conventional linings as well as reduced contamination by impurities from the lining material. Furthermore the graphite crucible can be changed easier than a ceramic lining and due to the low cross section a higher energy density can be maintained. The geometry of the crucible is illustrated in Figure 8.

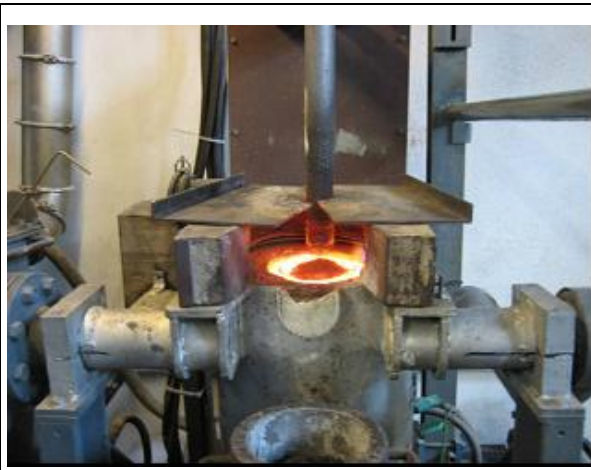


Figure 7: 100 kW AC electric arc furnace

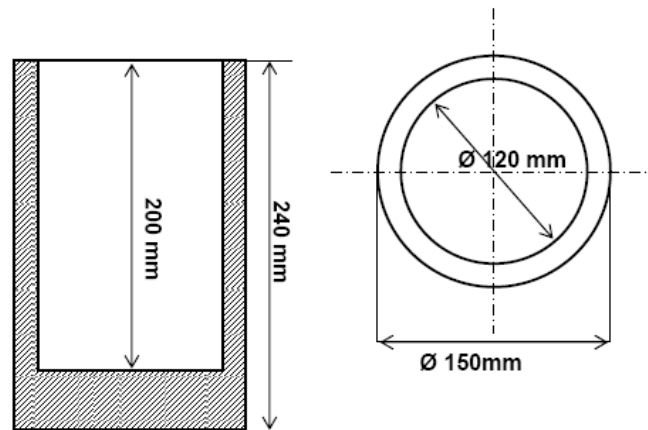


Figure 8: Geometry of the graphite crucible

4.3 Experimental test run

On the basis of the theoretical results 10 trials were conducted in the electric arc furnace (table 2). In order to control the reproducibility of the results almost every trial is repeated. The amount of lignite coke in each trial is stoichiometric to the total amount of oxides in the charged material. The first trials are carried out with an amount of silicon aiming on AlSi10 and AlSi30 alloy respectively. The Si-amount in the next trial is increased to 70 % and then lowered continuously down to 40 %. The trials V02a/b and V3a are conducted with an addition of steel scrap which was melted before charging of the pellets. The steel serves as collecting phase for the AlSi alloy.

At the beginning of the trials, the lining and the graphite crucible are heated in order to minimise the heat loss through the lining and in order to obtain the required process temperature. Heating was maintained by filling the crucible with coke and forming of an electric arc. The heating period is between 30 and 60 minutes after reaching a temperature between 1 400 and 1 500 °C. The first measurement of the temperature happens directly before starting to charge the crucible with pellets.



The measurement is carried out with an optical glass fibre, which is put in the crucible. The optical signal is converted and recorded in a pyrometrical measuring system.

Table 2: Overview of the conducted trials (“a, b, c”: repeating tests of same parameter set)

Trial	Targeted amount of silicon in emerging alloy	Mass of Iron scrap in g	Charged mass of pellets in g
V01	10 %	-	1 792
V02a	30 %	1 340	1 878
V02b	30 %	1 000	1 367
V03a	70 %	500	4 762
V03b	70 %	-	5 152
V03c	70 %	-	5 108
V04a	60 %	-	4 000
V04b	60 %	-	3 278
V05	50 %	-	3 360
V06	40 %	-	3 964

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Then the graphite crucible is filled completely with pellets in order to retain aluminium dust which originates from Al and AlO₂-evaporation at the bottom of the crucible. When passing through the pellet bed, aluminium containing gas components condensate and dust is adsorbed by the cooler unreacted pellets and circulate back to the bottom. Figure 9 illustrates the bed of the pellets in the crucible during the trial. Due to the chosen set up, the carbon in the pellets is exposed to high temperatures for some time, which leads to carbon losses by oxidation and therefore results in a sub-stoichiometric process.

In order to prevent sintering and bonding of the pellets during the process, the pellets were imposed with light pressure by means of a graphite stick once in a while. After finishing the trial, the electrode is removed from the crucible and the hollow space is filled with pellets. In this way the effect

of the porous bed structure is maintained until the reactions have completely stopped. After cooling down of the furnace the crucible is removed from the furnace and the formed product is analysed. The electrical data for the experiments resulted in about 0.5 A and 45 V respectively, according to a power of 22.5 kVA. With this power, short arc generation is possible and the efficiency of the pellets bed is increased.



Figure 9: Generated packed bed of pellets during the process

The measurement of the temperature is difficult in the required temperature interval. A continuous measurement isn't possible and it is hard to insert into the bed of pellets with the measuring device. That's why the measurement is carried out with the aforementioned optical glass fibre, which is pierced through the pellets.

5. Results

The separation of metal and unreacted material for the analysis and mass balance was difficult after the process due to the incomplete separation during the process. Metal drops were still present in the unreacted material and couldn't be always sorted out exactly. Table 3 illustrates the mass balances of the conducted trials. In every trial metal is formed except for V01, trial V04b had to be abandoned because of technical difficulties. In some trials the forming of funnels in the material bed is observed, formed by a strong emergence of CO during the reduction process. The gas passes through these funnels and dust from the bottom of the crucible is transported to the top and spattered which causes losses, as can be seen for trials V03 to V06. Always the formation of hollow spaces in the upper layer of pellets is observed, which collapse under the mechanical forces cause by the discontinuous stick treatment or on his own due to strong formation of gases and dust.



Table 3: Mass balance for the carbothermic reduction of Al_2O_3 - SiO_2 -pellets aiming on Al30-90Si70-10-alloys (“a, b, c”: repeating tests of same parameter set), pellet mixtures see chapter 3.1

Trial	Tem- perature in °C	Input		Output		loss Δm in g	theoretical amount of AlSi alloy in g
		Mass of pellets in g	Steel scrap in g	metal in g	unreacted material in g		
V01	-	1 792	-	-	-	-	538
V02a	1 958	1 878	1 340	1 412	1 770	36	652
V02b	1 723	1 367	1 000	928	1 363	76	472
V03a	1 800	4 762	500	790	660	4 294	1 590
V03b	1 718	5 152	-	496	1 197	3 459	1 563
V03c	1 833	5 108	-	139	2 220	2 749	1 550
V04a	1 642	4 000	-	290	1 214	2 496	1 200
V04b	1 713	3 278	-	-	1 398	-	1 198
V05	1 406	3 360	-	312	765	2 292	1 000
V06	1 600	3 964	-	214	1 131	2 619	1 162

The temperature which is measured discontinuously during the process varies strongly. Due to the measurement with an optical fibre which is pierced through the pellets, the exact position of the fibre isn't known. So it is not possible to conduct the trials reproducible with regard to the temperature. But the fact that metal appeared in the trials prove that the temperature was over 2 000 °C because the formation only takes place above this temperature range. That leads to the assumption that the position of the optical fibre was above the reduction zone.

Table 4 shows the analyses of the produced metal phases. The samples are taken by borings of the metal. The analysis for aluminium, iron and carbon is conducted by ICP and the analysis for silicon by RFA. A statement about the existing phases (presence as carbides or solute) is not possible with this method. Due to the inhomogeneity of the metal, the samples are analysed two or three times and the data in the table presents mean values. No analysis is conducted in trial 1 and 4 because of the aforementioned missing metal phase.

In the trials which were conducted with steel as collecting phase (V02a/b and V03a) the aluminium and silicon concentrations are extremely low. This is clearly apparent from parameter set V03a/b/c, where only the first trial is conducted with steel. From the following two trials of V03 it can be concluded that steel doesn't improve the yield for aluminium and silicon. But the analyses of the other trials show a big difference to the calculated values as well.



Table 4: Analysis of the metal after carbothermic reduction of Al_2O_3 - SiO_2 -pellets aiming on a Al30-90Si70-10-alloys and resulting recovery yields for Al and Si (“n.r.”: not relevant; “n.a.”: not available; “a, b, c”: repeating tests of same parameter set)

Trial	theoretical silicon content in the al- loy	Fe in %	Al in %	Al-yield in %	Si in %	Si-yield in %	C in %
V01	10 %	n.r.	n.a.	n.a.	n.a.	n.a.	n.a.
V02a	30 %	82.55	0.24	0.86	1.52	12.79	5.10
V02b	30 %	75.20	1.30	4.18	2.26	16.33	2.47
V03a	70 %	55.40	11.95	19.76	31.80	22.09	2.65
V03b	70 %	n.r.	20.50	21.66	46.70	20.72	6.60
V03c	70 %	n.r.	33.30	9.95	51.30	6.44	5.50
V04a	60 %	n.r.	25.10	15.06	36.80	14.33	9.95
V04b	60 %	n.r.	n.a.	n.a.	n.a.	n.a.	n.a.
V05	50 %	n.r.	11.00	6.86	27.00	16.55	16.00
V06	40 %	n.r.	7.50	2.30	31.60	14.23	12.00

A considerable influence of the dynamic bed structure is caused by the properties of the pellets. High amounts of silicon leads to sintering and forming of funnels in the bed and the temperature resistance of the pellets becomes more and more insufficient. In the lower parts of the bed no pellets are found anymore, they disintegrate and sintering of the resulted powder increases. As a consequence the gas permeability of the bed decreases. In figure 10 a scheme of the electric arc furnace with the assumed sequence of reactions and temperatures is shown. It illustrates the way of the components, which react and are formed in the crucible.

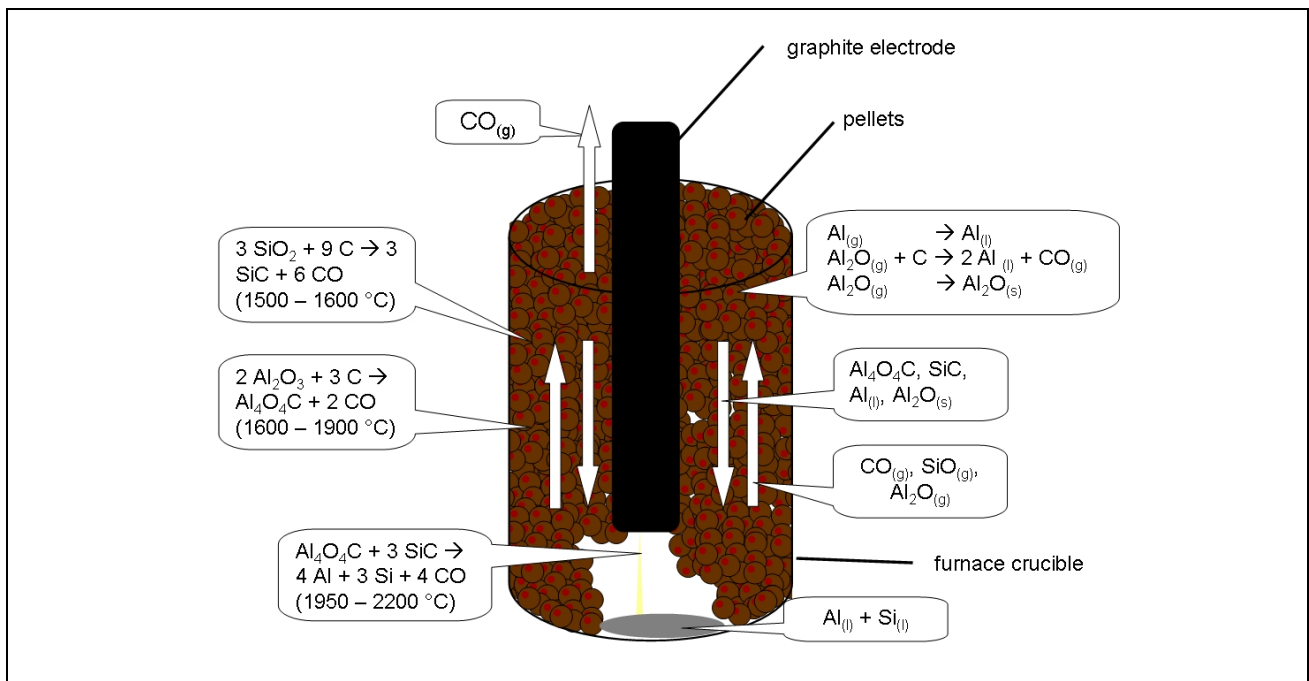


Figure 10: Scheme of the electric arc furnace with the assumed reactions and components

6. Conclusion and Recommendations for Next Steps

The conducted thermochemical calculation delivers a forecast into the reactions and the expected phase distribution during the process. However the calculation doesn't deliver information about the kinetics of the process. The experiments show that the implementation of the carbothermic reduction of used oxidic material is principle possible in order to produce an AlSi alloy. Metal was formed mostly but the metal yield in the trials must be considered as too low due to insufficient experience and the small scale equipment. The electric arc furnace seems to be a suitable technology, but apparently the bed of pellets could not be built high enough in the lab-scale-aggregate for a sufficient condensation of the evaporated aluminium and SiO. Furthermore material losses by dust carry over are excessively high which can be attributable to the fact that the strength of the pellets are too low. This has to be investigated and improved in further tests.

The development of the process conditions ("window") will be directed to achieve a metal phase with low impurities, especially carbon. Before conducting further tests in the EAF, investigation has to be done concerning the process conditions. In future equilibrium trials the optimal height of the pellet bed will be investigated in order to carry all gaseous metallic components. For this purpose the pellets were heated in a resistance heated furnace and the formed gaseous components are carried in pellet beds over the reaction zone with varying heights. The analysis of the products in these trials will help to understand and to identify the reactions which still aren't well known. Furthermore the measurement of the temperature has to be improved. The temperature of the process was



apparently sufficient for the reduction, but the measurement was not exactly enough and process control by temperature was not possible.

This preliminary study shows that thermochemistry and the thermokinetics of this process is quite complex. In order to understand and to control the process it is necessary to investigate more intensely these fundamentals. Nevertheless the carbothermic reduction of AlSi alloys from secondary oxide-based sources seems to be feasible, especially due to the fact, that this has been already conducted industrially for primary resources. The future aim is therefore to convert the existing (old or Russian) knowledge to this process in order to close one of the last gaps of the aluminium recycling loop.

7. Acknowledgement

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