RESEARCH ARTICLE

Phosphorus Gasification during the Reduction of basic Oxygen Furnace Slags in a Novel Reactor Concept

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The research presented in this publication focuses on the removal of phosphorus from basic oxygen furnace slags via the gas phase during carbo-thermal reduction in a bed of inductively heated graphite pieces. Its purpose is to evaluate the effect of the application of a novel reactor concept on the possibilities of phosphorus gasification. The slags are processed in two steps during the experimental trial: First, a silica source is added to reduce the basicity of the slag. In a second step, the actual reduction process is conducted at reaction temperatures of 1,623 K, 1,773 K and 1,923 K, respectively. The molten slag forms a film moving down the reactor with minimal contact time between iron (l) and phosphorus (g) so that a high amount of phosphorus gas can be removed from the reactor. The most important results are that more than 95% of the phosphorus contained in the slags could be reduced, roughly 85% of which were removed via the gas phase. Virtually iron-, chromium- and phosphorus-free slags are obtained. It is concluded that the application of the presented reactor concept based on thermodynamic evaluations potentially tackles the challenges posed by the reaction behaviour of phosphorus.

Introduction

The reduction of steelmaking slags (this paper will refer only to basic oxygen furnace slags – or LD slags – abbreviated as BOFS) is well investigated. These slags contain high amounts of valuable elements like iron, chromium, manganese in their oxidic form as well as phosphorus in compounds like $3CaO \cdot P_2O_5$ (C_3P). Phosphorus, in particular, is of immense importance to the production of food. It is used in fertilizers and cannot be substituted by any other element to fulfil vital functions in the human body. It is part of the genetic information – DNA – and involved in an energy supply mechanism involving adenosine triphosphate (ATP) [1]. It is included in the list of critical raw materials of the European Union together with its main primary resource phosphate rock [2]. The research effort dedicated to BOFS, however, aims at the recovery of valuable metals. Standard processes like electric arc furnaces are able to reduce iron, manganese and chromium oxides as well as phosphorus compounds to a high extent. The metal product obtained consists mostly of elemental iron, manganese, chromium but also of phosphorus and carbon [3,4]. For the direct reuse of the obtained metal product in the steelmaking process, phosphorus is the main limiting factor, because its accumulation in the process can impair the quality of the crude steel [5]. In a situation where the contact between phosphorus (g) and iron (l) is intense, phosphorus is enriched in the metal phase [6]. In the case of standard reduction furnaces, like electric arc furnaces, the presence of an iron bath and gaseous phosphorus enables this undesired interaction. Additionally, the temperature distribution is not sufficiently equal so that reactions cannot

be controlled [7]. Phosphorus is highly reactive and forms compounds with almost any other element. Therefore, reaction conditions have to be controlled well in order to maintain a certain product quality [8]. Phosphorus shall not be accumulated in a metal phase obtained via BOFS reduction. However, it has a certain affinity to phosphide formation (mainly with iron) and is soluble in liquid iron. Therefore, some research focuses on the addition of a refining step for the obtained phosphorus containing metal phase [9]. Similar research works on the partial reduction of BOFS so that metal oxides are reduced but phosphorus compounds remain in the slag matrix [10]. Only if phosphorus does not accumulate in the metal phase during complete BOFS reduction, high resource efficiency can be ensured.

In order to achieve the goals mentioned above, the subject of fully reducing BOFS and simultaneously removing phosphorus via the gas phase has been thoroughly investigated. Morita *et. al.* [11] describe an approach using microwave irradiation and carbon flux treatment. Nakase *et. al.* [12,13] followed a carbo-thermal reduction approach and investigated the influence of the slag composition. At high iron oxide contents, only a low amount of phosphorus has been removed from BOFS via the gas phase during the simultaneous reduction. The influence of the FeO activity on the phosphorus gasification has been described in great detail. It becomes clear by studying respective literature that the objective of gaseous phosphorus removal from BOFS during its reduction is only successful, if iron oxide contents are low. Nakase *et.al.* reduced slag with an FeO content of <2.5 m.-% (B2 = 1.0) at 1673 K and achieved a phosphorus removal rate of

roughly 55%. This is the highest value found in respective literature [13].

Similar problems related to phosphorus enrichment in a metal phase by the formation of Fe-P-compounds during reduction are reported by Matinde *et. al.* [14] to have occurred during the treatment of sewage sludge ashes. The mechanisms involved appear to be the same. However, reported research does not provide a sufficient approach to solve the problem.

One of the challenges often mentioned regarding the full reduction of BOFS is the stability of the calciumsilicate phases after reduction. If BOFS is merely partially reduced, phosphorus remains in the slag. The element is able to stabilise C_2S phases as β-C_2S. If a fully reduced slag is cooled too slowly, γ-C_2S is formed and a volume increase of more than 10% might occur [15]. With sufficient cooling rates this phenomenon has been prevented in the course of the research presented. These aspects, however, are not the main focus of the current publication.

Phosphorus contents in slags are often stated as P_2O_5 contents in mass fractions (m.-%). However, the main phosphorus compound in BOFS is calcium phosphate (C_3P) [16]. In order to determine the required reduction potential, it must be considered that the position of phosphorus in a Richardson-Ellingham is completely different from that of P_2O_5 (s. supporting information). The following reduction reaction must be considered (Equation (1)) [17]:

$$(3\ CaO \cdot P_2O_5) + 3\ (SiO_2) + 5\ [C] \\ \rightleftharpoons 3\ (CaSiO_3) + \{P_2\} + 5\ \{CO\} \quad (1)$$
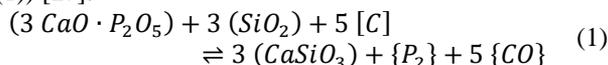
Richardson-Ellingham diagrams normalise everything to 1 mole of oxygen. In order to compare the required reduction potential to that of metal oxides, the following reaction has to be analysed (Equation (2)) [18]:

$$\frac{6}{5}\ CaO + \frac{2}{5}\ P_2 + O_2 \rightleftharpoons \frac{2}{5}\ (3\ CaO \cdot P_2O_5) \quad (2)$$
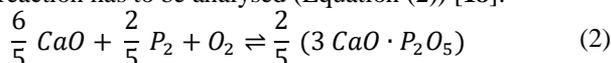
The involvement of silica also influences the reduction behaviour of C_3P (s. supporting information). The production of gaseous phosphorus according to Equation (1) is shown in **Fig. 1** [19].
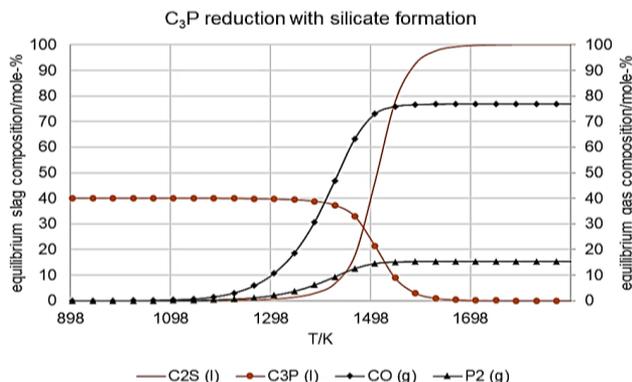

Fig. 1. Direct reduction of C_3P considering the involvement of SiO_2 [19].

As soon as elemental phosphorus exists, its relatively slow diffusion rates in slag and metal inhibit mass transfer [20]. Upon solidification, phosphorus dissolved in the liquid metal phase (mostly iron) forms phosphides. The high reduction temperatures inhibit the phosphide formation rates (s. supporting information). A slight under-pressure and the high temperatures support the desorption of elemental phosphorus from the liquid metal phase into the gas phase. The interface is as high as possible due to the packed bed of graphite pieces. The equilibrium is a simple distribution between phosphorus that is absorbed in the liquid metal and elemental, gaseous phosphorus as described in Nakase *et. al.* [13].

From these main reaction mechanisms and transport rate considerations, requirements for a reactor can be deducted:

- large surface area (ideally a film)
- direct heat input (endothermal reactions, ideally induction)
- thin slag/metal films (ideally a high temperature packed column)
- gas removal for minimum contact time between elemental phosphorus and metal

The reactor concept used for the experiments conducted tackles the problems regarding phosphorus-iron-reactions. Their contact is limited by providing a thin molten slag film on the surface of graphite pieces. Mass transport distances are therefore kept short. In a pilot-scale plant, an induced draft fan ensures the continuous suction of reaction gases from the reactor. In the lab-scale plant used for the research presented in this paper, an atmosphere alteration and a gas suction are not required due to batch operation. Heat is provided by induction directly beneath the individual particle surfaces, so that the endothermic reduction reactions are permanently supported and heat transfer is eliminated as a constriction. The functionality of the concept has been proven by the treatment of sewage sludge ashes, which also contain high amounts of iron oxides and phosphates [21]. The pilot-scale reactor is schematically shown in **Fig. 2** [22].
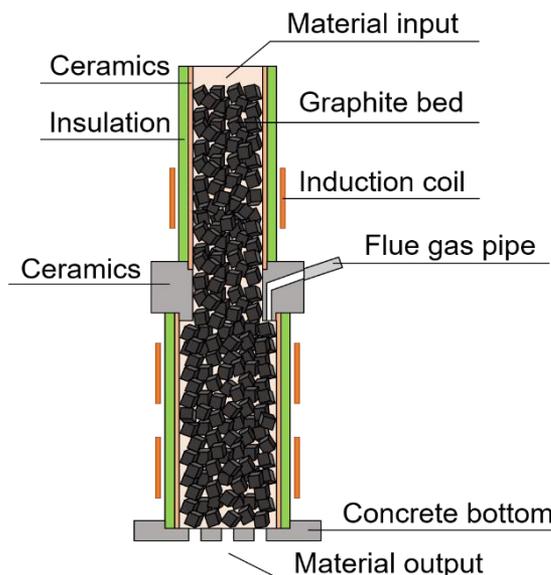

Fig. 2. Inductively heated bed of graphite pieces [22].

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In previous publications, the results have been partially presented [**19,22**]. Meanwhile, further analyses, mineralogical considerations, thermodynamic analyses and additional experiments have been added to underline the impact of the current findings. Phosphorus gasification rates with the application of the reactor concept described above have exceeded the state of knowledge by investigating the desorption equilibrium and the distribution of phosphorus between liquid metal (dissolved) and gas phase (elemental) and designing a reactor based on the thermodynamic behaviour of phosphorus in said application. The reaction mechanisms involved are depicted schematically in **Fig. 3** [**19**].

$$2\,(3\,CaO \cdot P_2O_5) + 3\,(SiO_2) + 10\,C \rightleftharpoons 3\,(2\,CaO \cdot SiO_2) + 2\,\{P_2\} + 10\,\{CO\}$$

$$(Me_xO_y) + y\,C \rightleftharpoons x\,[Me] + y\,\{CO\}$$

$$C + \{CO_2\} \rightleftharpoons 2\,\{CO\}$$

$$(FeO) + \{CO\} \rightleftharpoons [Fe] + \{CO_2\}$$

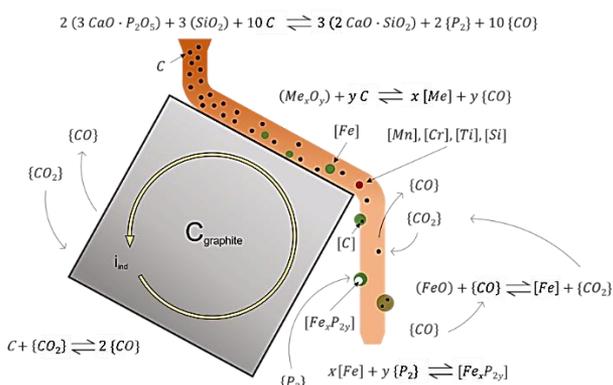$$x[Fe] + y\,\{P_2\} \rightleftharpoons [Fe_xP_{2y}]$$

Fig. 3. Schematic depiction of the reaction mechanisms for the reduction of basic oxygen furnace slags in the InduRed reactor considering phosphide formation [19].

Experimental

In order to identify a suitable point of operation for the continuous treatment of BOFS, the slags were altered in basicity (B2 is used to describe basicity in this publication and is defined as the ratio of CaO to SiO_2 as mass fractions). As a silica source, pure quartz sand (QS) as well as blast furnace slag (BFS) were used. **Table 1** shows the slag mixtures that were tested and **Table 2** shows the composition of each material. The total mass of each mixture was 2.5 kg.

Table 1. Investigated mixture compositions of BOFS, BFS and QS.

Mixture no.	B2 [-]	BOFS [g]	BFS [g]	QS [g]
1	1.5	1,269	1,231	-
2	2.5	2,174	326	-
3	1.5	2,167	-	333
4	2.5	2,407	-	93

Table 2. Input material composition for BOFS, BFS and QS.

Material	Fe [m.-%]	SiO_2 [m.-%]	Cr_2O_3 [m.-%]	MnO [m.-%]
BFS	0.50	37.88	-	1.47
BOFS	19.44	12.40	0.40	4.40
QS	0.18	93.21	-	-

Material	CaO [m.-%]	MgO [m.-%]	Al_2O_3 [m.-%]	P_2O_5 [m.-%]
BFS	34.76	10.51	10.78	0.01
BOFS	40.00	7.10	1.50	1.10
QS	0.10	-	3.61	-

The choice of the slag basicity is based on a prior evaluation of potential points of operation. Low B2 values mean high additive amounts, while high B2 values require more energy input and high reaction temperatures due to limited slag flowability. The possible spectrum was determined to lie between 1.0 and 3.0, with 1.5 and 2.5 chosen in order not to start the investigation at the endpoints of that spectrum.

The modification step is separated from the reduction step so that the formation of a new, homogeneous slag can be assured. The smelting of BOFS and the respective additives was conducted in a ceramic crucible that was heated by a graphite ring positioned around it. Thereby, the same induction furnace as for the reduction experiments could be used. The furnace is shown in the supporting information and the respective experimental reduction setup can be seen in **Fig. 4**. In order to prevent the formation of a liquid bath of iron, through which gaseous phosphorus has to move after reduction, a bed of graphite cubes is used inside a ceramic crucible.
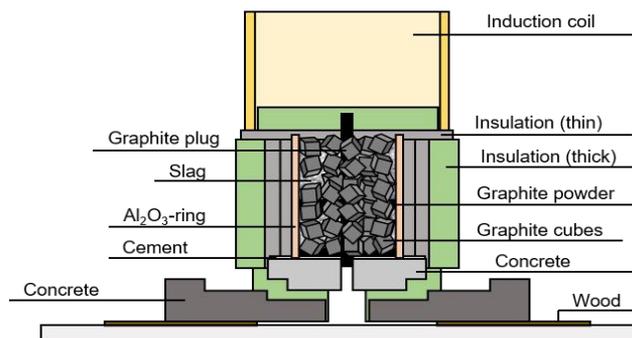

Fig. 4. Experimental setup for reduction experiments.

The cubes are roughly 2.5 cm in length and around 60 cubes are filled into a ceramic ring that is 12 cm in height and has an inner diameter of 14 cm. The ring is attached to a concrete bottom with refractory mortar. After insulation, the reactor is positioned below the induction coil of a specially adapted induction furnace, reaching some 5cm into it. 0.5kg of each tapped slag mixture (of the 2.5kg produced each as described above) were used for the reduction experiments and 8 m.-% of carbon powder were added.

The smelting step occurred at 1,823 K each. For the reduction and the identification of a suitable point of operation, three different temperatures were applied: 1,623K (low, abbreviated as l), 1,773K (medium, abbreviated as m) and 1,923K (high, abbreviated as h). Whenever tapping of the new slag mixture from the smelting step (1-4, s. Table 1) was possible (limited by tapping hole diameter of 8 mm and viscosity), the slag was reduced. Of these mixtures, the reduced slags of experiments 1 h, 3 m and 3 h (each indicating the mixture and the reduction temperature) could be tapped. Due to the full reduction and the decrease of the FeO-amount, the flowability of the slags decreases during the process and must not fall below a certain value for the reactor concept to work.

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Results and discussion

In the following paragraphs, the results are presented and evaluated.

Mineralogical evaluation

SEM and XRD analyses show that the products of the smelting step are homogeneous mixtures of the respective compounds. When QS is added to the BOFS, spinels are formed, in which chromium can be found. At a basicity of 2.5, C_2S exists as Larnite. If BFS is added, iron is also found in spinels and alkaline compounds form feldspar. A number of other void phases occur, which contain phosphorus. The number of phases is naturally higher, if the amount of impurities is higher (as is the case in a mixture of BOFS and BFS at B2=2.5). For the reduction experiments, only a fraction of the smelting products is used. Therefore, it is important to confirm the homogeneity of the slag products. However, regardless of the slag mixture phase composition from the smelting step, the reduced slags show a high similarity, regardless of their origin. The main compound is always Melilite, with Merwinite and Monticellite found as well. The higher the temperature is and especially when QS is used for the basicity alteration, the higher the reduction degrees are. With full reduction, less Merwinite occurs. Void phases contain minute amounts of phosphorus and chromium. Manganese, while reduced, forms MnS with residual sulphur and remains in the slags mostly as such. Only a minute amount of manganese is found in an oxidic form due to its reduction behaviour. The mineralogical analysis shows a good correlation with the results obtained from the ICP-MS. It can be concluded from the similarity of the product slags – regardless of the silica source used – that the reduction process can be completed. This is important in order to detect potential kinetic limitations and to further deduce retention rimes for a continuous process and future research.

Reduction degree evaluation and phosphorus gasification

The metal amount is quite low and due to the quick cooling after tapping and the high surface tension of the metal alloy forming, the metal phase exists in the form of small spheres after the reduction. They are spread out within the slag matrix and on the cubes' surface and reach a diameter of up to 5 mm in some cases. Mostly, they are <1 mm in circumference. Therefore, a sole gravimetric determination of the metal phase mass is impossible in most cases.

In order to tackle this problem, the metal phase (a sample of spheres from all over the product mass) and the slag phase are analysed by ICP-MS separately. To ensure that no metal is analysed together with slag and the other way around, the sample is crushed, separated by a magnet and all particles for analysis are weighed and compared in density. Additionally, calcium is analysed in the metal phase, which could only stem from a slag particle. All the values shown in tables and figures are corrected in case slag was analysed together with the metal, which was hardly ever the case and if it was the correction changed

element contents only in the third digit after the decimal point if stated in m.-%. Based on the iron content, iteration is used to determine the slag and metal phase mass. A comparison to the calculated fraction masses shows good correlation. It can be seen in **Table 3**.

Table 3. Correlation between weighed, calculated and analysed metal mass.

Evaluation method	ICP-MS	weighing	calculated
metal without P [g]	89.88	-	92.97
P in metal [g]	0.34	-	-
total [g]	90.22	92.5	-

Table 4 shows the contents of Fe, Cr, Mn and P in the metal and slag phases.

Table 4. ICP-MS analysis results for the metal and slag phases obtained in the reduction experiments.

species in metal	experiment 1h	experiment 3h	experiment 3m
Fe [m.-%]	75.40	93.10	92.40
P [m.-%]	0.61	0.38	0.37
Cr [m.-%]	1.02	0.77	0.63
Mn [m.-%]	1.23	4.49	1.32
Ca [m.-%]	0.12	0.15	0.33
S [m.-%]	-	0.03	-
species in slag	experiment 1h	experiment 3h	experiment 3m
Fe [m.-%]	0.31	0.11	1.78
P [m.-%]	0.01	0.00	0.04
Cr [m.-%]	0.05	0.01	0.08
Mn [m.-%]	1.97	2.02	3.25
Ca [m.-%]	23.50	24.70	23.10
S [m.-%]	-	0.71	-

It can be seen that the slag is basically chromium-, iron- and phosphorus-free. This correlates well with the mineralogical analyses. The reduction degree is calculated by assuming that the elements in the slag are bound in an oxidic form in the slag matrix and occur in their elemental form in the metal phase. They represent the ratio of metallic output to oxidic input and are shown for Fe, Cr, Mn and P in **Fig. 5**.
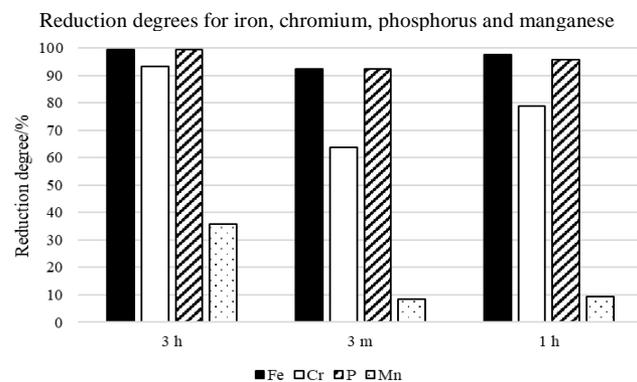

Reduction degrees for iron, chromium, phosphorus and manganese
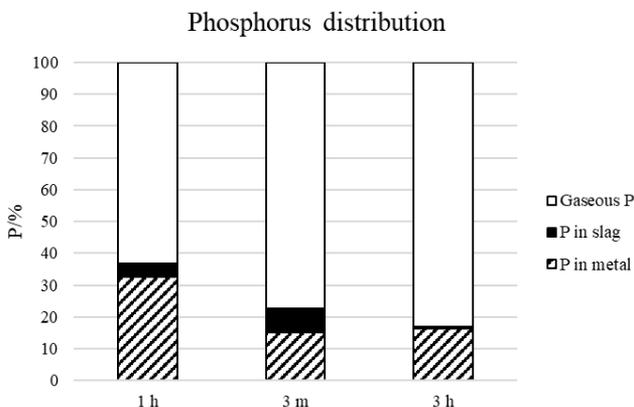
Fig. 5. Reduction degrees obtained by treatment in the inductively heated graphite bed reactor [19].
3 h: mixture of basic oxygen furnace slag and quartz sand (B2 = 1.5), T = 1,923 K
3 m: mixture of basic oxygen furnace slag and quartz sand (B2 = 1.5), T = 1,773 K
1 h: mixture of basic oxygen furnace slag and blast furnace slag (B2 = 1.5), T = 1,923 K

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With metal and slag product phase fractions determined and elements analysed, the phosphorus balances can be calculated. The P found in slag and metal subtracted from the input amount of phosphorus in each mixture is assumed to have left the reactor in its gaseous form. The P-balances are depicted in **Fig. 6**.

The obtained metal phases also contain silicon. At 1,923 K, SiO_2 is reduced (s. Richardson-Ellingham diagram). A thermodynamic investigation of the behaviour of other elements than the ones discussed will be provided in future publications.

Fig. 6. Phosphorus degree obtained in inductively heated graphite bed reactor [19].
3 h: mixture of basic oxygen furnace slag and quartz sand (B2 = 1.5), T = 1,923 K
3 m: mixture of basic oxygen furnace slag and quartz sand (B2 = 1.5), T = 1,773 K
1 h: mixture of basic oxygen furnace slag and blast furnace slag (B2 = 1.5), T = 1,923 K

The amount of 83% in experiment 3h is especially remarkable as it exceeds values reported in respective literature and is to be validated in future experiments. The reactor concept facilitates the desired reactions according to the thermodynamic evaluation described above.

Comparing the results obtained by the use of different silica sources, it can be seen that both additives show the potential to serve as a suitable silica source for the desired process. In the respective experiments, the phosphorus gasification was possible to a high extent. The results with pure quartz sand were slightly better. However, in the future the number of experiments conducted has to be increased to verify these results. Not introducing a number of additional slag phases by using BFS to the process might have a positive effect on the product slag quality. However, it can be a cost-efficient additive.

As has been mentioned above, Mn cannot be fully removed in some of the experiments. XRD has identified MnS within the slag matrix. Residual S might bind the reduced Mn, so that the reduction degree is distorted (S was also analysed and found by ICP-MS analysis to an extent that it can react with roughly 60% of the Mn present, s. **Table 4**). It seems that the slag can bind Mn to a certain extent and better than chromium. Because of the low Cr amounts, analysis accuracy plays a big role. By treating

synthetic slags containing varying amounts of MnO and Cr_2O_3, the reduction behaviour of MnO will be further investigated in the future. Additionally, the carbon uptake in the alloy has to be considered. Follow-up research will also include increasing the number of experiments, further analyses and creating energy balances of the suggested recycling process.

Conclusion

The novel reactor concept is potentially able to tackle existing problems in the field of BOFS reduction and phosphorus gasification.

1. Reduction rates of over 95% for iron and phosphorus as well as over 90% for chromium have been achieved.
2. The phosphorus could be gasified to an extent of up to 83%, exceeding the state of knowledge.
3. BFS has shown to be a suitable silica source for the BOFS alteration needed for the conduction of the described process.

Thermodynamic and transport investigations have shown that phosphorus needs high temperatures, short mass transport distances and a large surface area to be transferred into the gas phase at high rates. The determined equilibrium distribution is also influenced by the simultaneous formation of phosphides. High reaction temperatures benefit the inhibition of Fe-P-compound formation. The experiments designed, conducted and evaluated corroborate these hypotheses.

Future research will focus on the verification and reproduction of the phosphorus removal rates as well as on the further improvement of the metal quality. The manganese recovery rates, the carbon uptake, the decrease of the phosphorus fraction in the metal phase, and the modification of the process route are and will be aspects of current and future research activities.

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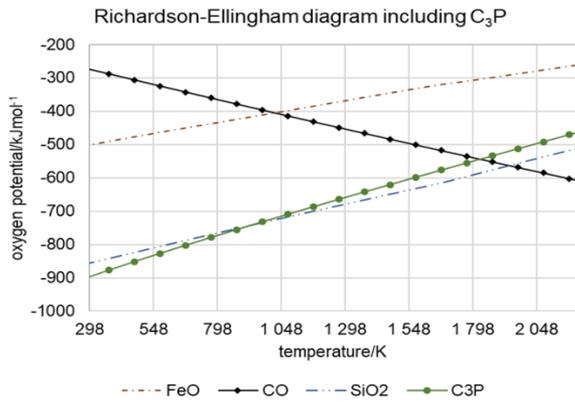
Supporting information

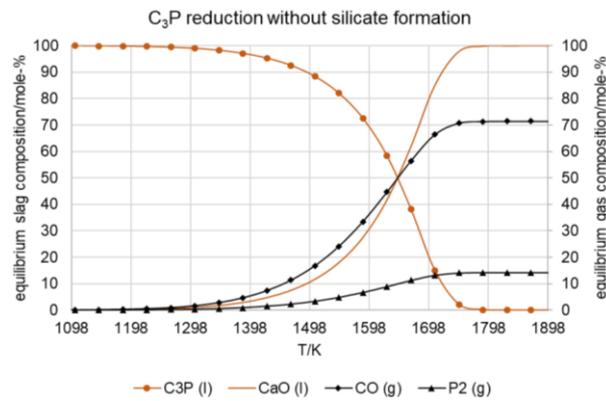**Fig. 7.** Position of C_3P in a Richardson-Ellingham diagram [19].

Fig. 8. Direct reduction of C_3P not considering the involvement of SiO_2 [19].
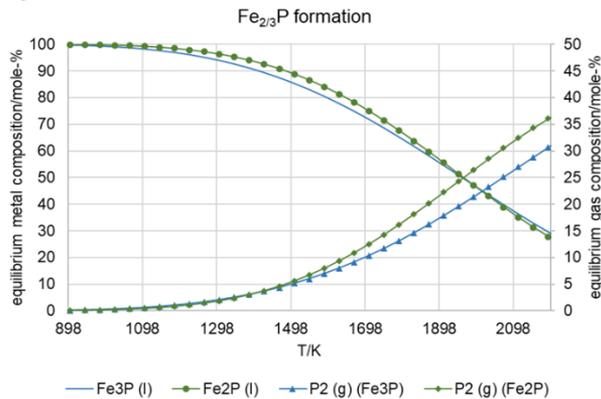

Fig. 9. $Fe_{2/3}P$ formation over increasing temperature [19].
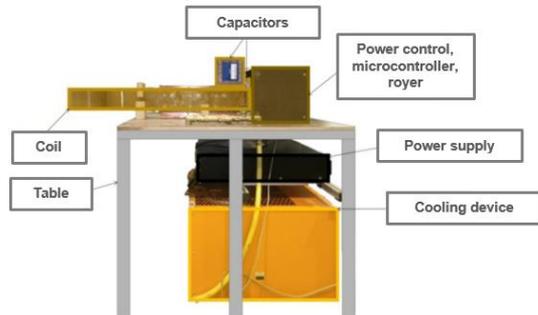

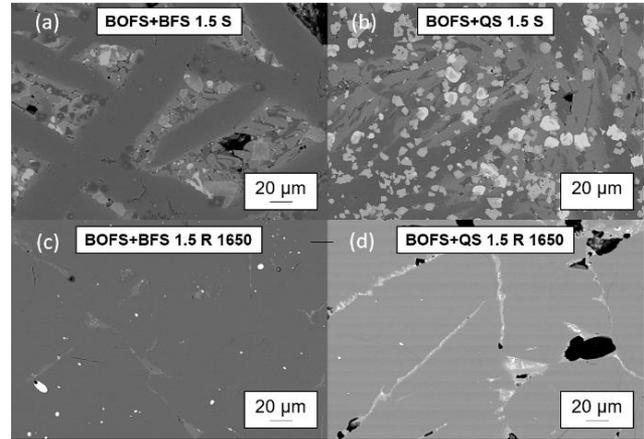**Fig. 10.** Induction furnace used for smelting and reduction experiments.

Fig. 11. SEM images of produced slag samples: (a) BOFS+BFS, B2=1.5, smelting step (b) BOFS+QS, B2=1.5, smelting step (c) BOFS+BFS, B2= 1.5, reduction at 1,923 K (d) BOFS+QS, B2=1.5, reduction at 1,923 K.

Authors Biography

Dr. Christoph Ponak works as a researcher at the Chair of Thermal Processing Technology of the Montanuniversitaet Leoben, Austria. He studied Industrial Environmental Protection and Process Technology and in his doctoral thesis focused on high-temperature recycling processes involving the reaction behaviour of elemental phosphorus. He is currently working on the development and improvement of processes for sewage sludge ash, steelmaking slag and Li-ion battery recycling.

Graphical Abstract
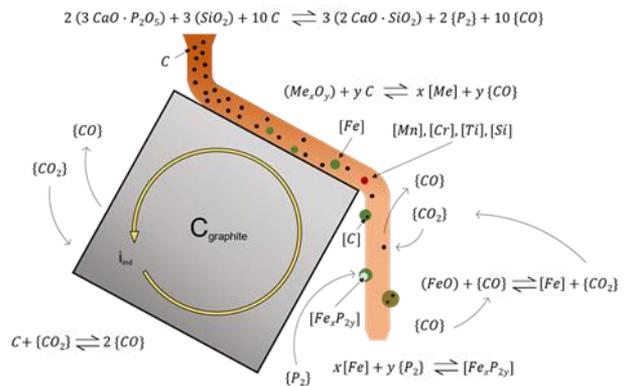
The reaction behaviour of phosphorus poses a lot of challenges regarding the recycling of basic oxygen furnace slags. If the slags are reduced completely in order to recover iron, manganese and chromium, phosphorus must be removed via the gas phase by influencing the desorption equilibrium and providing short mass transport distances. The InduRed reactor serves as a "high temperature packed column" and has proven successful in tackling the mentioned challenges.