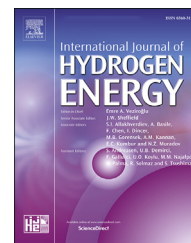


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Review Article

A review on the fundamentals of hydrogen-based reduction and recycling concepts for electric arc furnace dust extended by a novel conceptualization



Ulrich Brandner^{*}, Juergen Antrekowitsch, Manuel Leuchtenmueller

Chair of Nonferrous Metallurgy, Montanuniversität Leoben, Franz-Josef-Strasse 18, 8700 Leoben, Austria

HIGHLIGHTS

- Identified hydrogen as a high-potential alternative reducing agent for electric arc furnace dust (EAFD).
- Elaborated the current state of the research about hydrogen in the field of EAFD recycling.
- Presented currently available process concepts for EAFD.
- Outlined an innovative process conceptualization for the processing of EAFD with hydrogen.

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ABSTRACT

The European Green Deal aims to make Europe climate neutral by 2050. This circumstance requires the metallurgical industry to move from carbon-based processes to hydrogen application. This work aims to summarize the current state of the research on thermodynamics and reaction kinetics in the scope of hydrogen-based reduction for Electric Arc Furnace Dust and present already existing and theoretically possible recycling processes using hydrogen. Thermodynamic calculations suggest three possible approaches to treat Electric Arc Furnace Dust using hydrogen as a reducing agent: Full reduction of iron and zinc oxide, selective reduction of iron oxide, and selective reduction of zinc oxide. In general, hydrogen can replace carbon seamlessly, leading to higher reaction kinetics but also to higher processing costs due to the high hydrogen price currently. However, zinc extraction from Electric Arc Furnace Dust allows for innovative concepts such as hydrogen recovery by reoxidation of gaseous zinc with water. In combination with selective zinc oxide reduction, this approach results in a recycling process with a low hydrogen consumption. However, additional high-quality research is necessary to provide a better data basis for a detailed economic view.

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^{*} Corresponding author.

E-mail addresses: ulrich.brandner@unileoben.ac.at (U. Brandner), juergen.antrekowitsch@unileoben.ac.at (J. Antrekowitsch), manuel.leuchtenmueller@unileoben.ac.at (M. Leuchtenmueller).
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Introduction

In the last century, the share of carbon dioxide in the atmosphere has reached the highest level since the beginning of the recordings [1]. The Paris agreement and its' ambitious goals towards keeping the global temperature rise this century well below two degrees Celsius above pre-industrial levels confront industrialized countries all over the world with a severe challenge to reduce carbon dioxide emissions [2].

Right now, the steel industry causes 7.2% of the total anthropogenic carbon dioxide emissions. The total steel demand is forecasted to increase by one-third until 2050, leading to a further increase in carbon dioxide emissions [3]. The higher scrap availability results in more steel production via the Electric Arc Furnace route forecasted to more than double from 517 in 2019 to 1200 million tons until the middle of the century [4,5]. The melting of steel scrap in an Electric Arc Furnace generates 15–25 kg Electric Arc Furnace Dust (EAFD) per ton of steel [6]. Thus, the generated dust amount is now 8 million tons per year and will increase to at least 18 million tons by 2050. EAFD consists of metals like iron, zinc, lead, sodium, cadmium and nickel in oxidic form with a complex morphology. The main constituents are iron and zinc and occur as magnetite (Fe_3O_4), hematite (Fe_2O_3), zinc ferrite (ZnOFe_2O_3) and zincite (ZnO) [7,8]. The ratio between zinc ferrite and zincite depends on the ratio between zinc and iron and the conditions in the off-gas system of the electric arc furnace [7]. The share of zincite increases with higher zinc levels [7]. Additionally, EAFD contains chlorides, fluorides and slag building components like silicon dioxide (SiO_2) and calcium dioxide (CaO) [7]. Most developed countries classify EAFD as hazardous waste (Europe: European Waste Catalogue 10 02 07, United States: Environmental Protection Agency K061* and Canada: Ministry of the Environment 143H). However, it contains up to 43% of valuable zinc [9–12]. Thus, processing of EAFD in a treatment process is required from an ecological point of view and economically attractive. The state-of-the-art EAFD-recycling processes are based on

carbon-based reduction methods like the Waelz process, contributing to the carbon dioxide emissions of the steel industry [13]. One approach towards decreasing carbon dioxide emissions is the substitution of carbon-based reducing agents with hydrogen. Different process concepts based on hydrogen have been presented. Nevertheless, no hydrogen-based concept has reached an industrial scale application so far. The main reason for this circumstance is the current dominance of the Waelz process arising from low operating costs due to the application of cheap carbonaceous reducing agents and the energetically efficient operation mode. Additionally, the implementation of novel hydrogen-based processing technologies suffers from high capital expenditures [14]. Moreover, the availability of hydrogen is not capable comprehensively right now [15].

However, on the one hand, carbon dioxide certificates are likely to rise. On the other hand, production costs for hydrogen are forecasted to decline, making hydrogen-based reduction a viable alternative in the scope of EAFD recycling [14]. On top of that, societal pressure to fulfill the targets mentioned initially forces the switch to hydrogen-based metallurgy. Thus, this study was conducted to outline the status quo of the application of hydrogen as a reducing agent in terms of EAFD recycling. Its' objective is to give an overview of the currently available literature and process concepts. In addition, a novel low hydrogen-consumption conceptualization for EAFD recycling is elaborated.

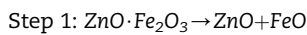
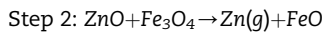
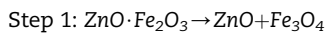
Fundamentals of the hydrogen-based reduction of EAFD

This chapter compares the thermodynamics of hydrogen and carbon-based reduction for iron and zinc oxides and discusses qualitatively how kinetic aspects change if hydrogen substitutes carbon-based reduction. Thereby, the process concepts described subsequently can be categorized from a thermodynamic point of view and no kinetical limitations

arise in most parts of EAFD recycling when hydrogen substitutes carbon.

Thermodynamic aspects

Iron and zinc are the main constituents of EAFD present as oxides like magnetite (Fe_3O_4), hematite (Fe_2O_3), zinc oxide (ZnO), or zinc ferrite ($\text{ZnO} \cdot \text{Fe}_2\text{O}_3$) [7]. EAFD usually aims to separate iron (Fe) and zinc (Zn) to produce a zinc-enriched fraction. This is generally achieved by the reduction of iron and zinc oxide at increased temperatures. In the high-temperature processes zinc oxide reacts to gaseous metallic zinc separable from the reduced solid phase via the off-gas. The following reduction paths are postulated for the reduction of zinc ferrite with hydrogen [16–18]:



When hydrogen is already applied during heating, zinc ferrite initially dissociates into zinc oxide and hematite [16,17]. Whereas zinc oxide is directly reduceable to zinc, magnetite is reduced to wustite (FeO), which is then further reduced to metallic iron [16,17]. If hydrogen is only applied when the respective reduction temperature is reached, the reduction of zinc ferrite was described to run directly to wustite and zinc oxide. Subsequently, wustite is reduced to iron and zinc oxide to zinc [18].

The application of hydrogen as a reducing agent results in an atmosphere comprising of hydrogen and water vapor. Fig. 1 illustrates the thermodynamic stability regions of zinc, iron and the respective oxides depending on the temperature and gas composition. Fig. 1a shows the influence of the ratio between hydrogen and water vapor, Fig. 1b the influence of the ratio between carbon monoxide and carbon dioxide. This allows for a direct comparison of carbon-based reduction methods like the Waelz kiln with hydrogen-based approaches.

The lines separating the stability fields of the respective oxides and metals were calculated from the reduction reactions, which are listed below. The activities of solids were set to 1, only the partial pressures of the gaseous components were considered for the calculation of the stability regions in Fig. 1.

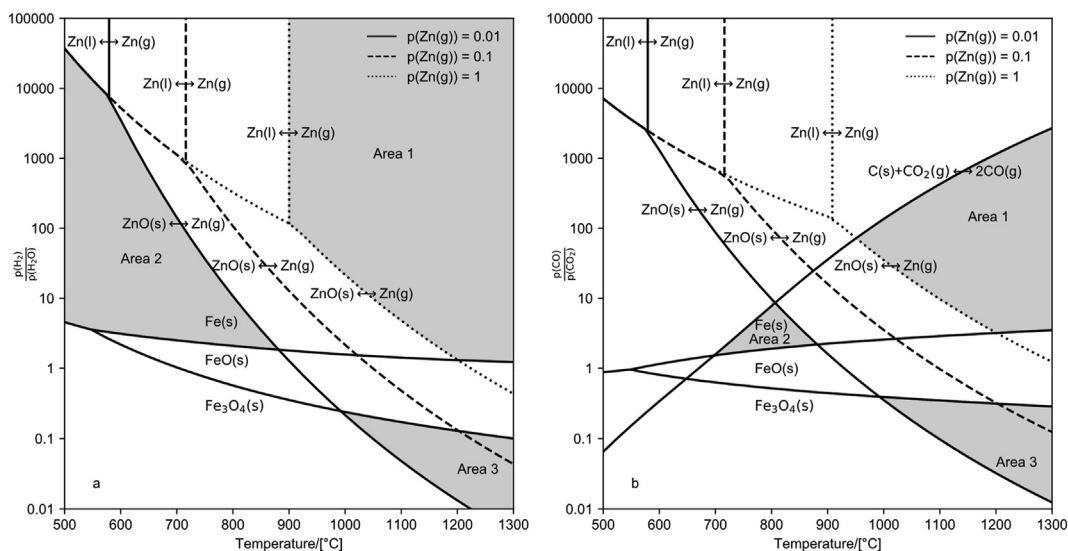
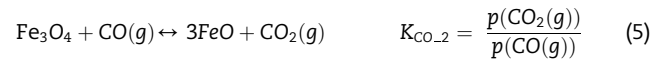
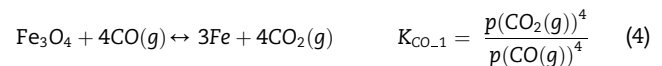
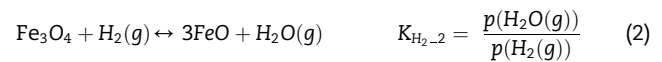
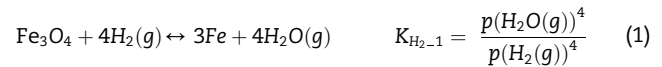
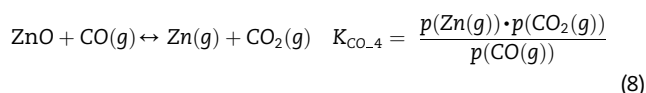
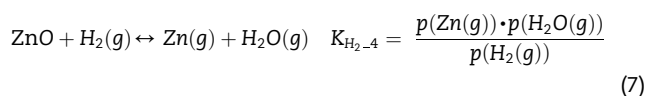


Fig. 1 – a: $\text{Fe(s)}-\text{FeO(s)}-\text{Fe}_3\text{O}_4(\text{s})-\text{Zn(g)}-\text{Zn(l)}-\text{ZnO(s)}-\text{H}_2(\text{g})-\text{H}_2\text{O(g)}$ stability state diagram b: $\text{Fe(s)}-\text{FeO(s)}-\text{Fe}_3\text{O}_4(\text{s})-\text{Zn(g)}-\text{Zn(l)}-\text{ZnO(s)}-\text{C(s)}-\text{CO(g)}-\text{CO}_2(\text{g})$ stability state diagram [19].



Reactions 1, 2 and 3 describe the iron oxide reduction applying hydrogen as the reduction agent, reaction 4, 5 and 6 describe it for carbon monoxide. Reactions 7 and 8 represent the reduction of zinc oxide with hydrogen and carbon monoxide, respectively. Reaction 9 outlines the Boudouard-equilibrium.

The reduction of magnetite to iron is feasible in the entire temperature range as long as the ratio of hydrogen to water vapor or carbon monoxide to carbon dioxide is high. Up to 570 °C magnetite reacts directly to iron. Above this temperature, magnetite forms the intermediate phase wustite, which then reacts to iron. While the reduction potential increases with higher temperature using hydrogen, it decreases for carbon monoxide. The stability region for metallic zinc shows a higher temperature dependency. The stability regions enlarge with increasing temperature for both reduction agents. A decrease of the gaseous zinc partial pressure (Equations (7) and (8)) further increases the stability region for zinc.

Fig. 1a can be separated into the following three reduction zones:

- Area 1: Complete iron and zinc oxide reduction is possible at a high temperature and a high ratio between hydrogen and water vapor in the gas.
- Area 2: Selective iron oxide reduction is possible at a low temperature and a ratio between hydrogen and water vapor of at least approximately 10 in the gas.
- Area 3: Selective zinc oxide reduction is possible at a high temperature and a ratio between hydrogen and water vapor below approximately 0.1.

Fig. 1b additionally includes the Boudouard-equilibrium, which indicates the temperature-dependent equilibrium concentration of carbon, carbon monoxide and carbon dioxide. The Boudouard-line limits the reduction areas 1 and 2. Above the Boudouard-line, carbon monoxide decomposes into carbon and carbon dioxide. Thus, a reducing gas composition exceeding the equilibrium line in Fig. 1b is thermodynamically unstable and the reaction to iron or zinc cannot occur. This phenomenon mainly restricts the reducing potential of carbon monoxide at a relatively low temperature. In contrast, hydrogen does not have a similar restrictive phenomenon arising from the Boudouard-equilibrium.

Kinetic aspects

The state-of-the-art process to treat EAFD requires agglomeration and mixing with carbon. The reduction itself runs at 1200 °C via carbon monoxide, which reacts with zinc oxide to

carbon dioxide. Carbon dioxide and carbon then react via the Boudouard-reaction back to carbon monoxide. Therefore, the reaction system for hydrogen- and carbon-based processes is of solid-gaseous type. At 800 °C the reduction of zinc ferrite with hydrogen is ten times faster than with carbon monoxide [18,20].

Different rate-limiting steps, e.g., gaseous or solid diffusion, chemical reaction or nucleation can occur. The following parameters influence the reduction kinetics:

- Temperature.
- Partial pressure of hydrogen.
- Pellet size.
- Morphological composition of the pelletized material.

Increasing the process temperature leads to a higher reduction rate [18]. The temperature additionally determines the rate-limiting step of the reaction [21]. In the temperature range of 550–650 °C the reduction is controlled by nucleation [21]. Between 700 °C and 800 °C a mixture of nucleation and diffusion is dominant due to iron forms a crust above 700 °C hindering the diffusion of the reducing gas into the pellet [21]. Between 850 °C and 950 °C sintering effects lead to decreased pore volume [21]. Thus, diffusion becomes rate-limiting [21,22].

Tong and Hayes describe that a decreased hydrogen partial pressure (e.g., by enriching the gas with nitrogen) favors the generation of a dense iron layer on the intermediate wustite surface. This phenomenon occurs because the lower hydrogen partial pressure decreases the reduction rate of the wustite to iron. Thus, the wustite reduction occurs uniformly and a dense iron layer instead of a dendritic structure covers the intermediate wustite layer. The direct access of the reducing agent to the wustite is hindered. The ongoing reduction of wustite to iron can only proceed via the solid diffusion of oxygen through the dense iron layer. Thus, the reduction rate is decreased with a decreasing hydrogen partial pressure [18].

Junca et al. showed in investigations with pelletized EAFD and zinc ferrite that the reduction rate decreases with larger pellet sizes ranging from 13 mm to 7 mm. The reason is the required diffusion from the surface to the core. At a higher temperature, the rate-limiting mechanism shifts from nucleation to diffusion. Thus, the influence of the pellet size on the reduction rate is even more pronounced at a higher temperature [22,23].

The morphological composition of the EAFD pellets influences the reaction kinetics. The presence of 1% calcium oxide (CaO) in zinc ferrite slightly raises the reduction rate, because calcium oxide fragments the pore structure of the intermediate wustite layer [18].

Process concepts

Fig. 2 gives an overview of process concepts and divides them into three approaches according to Fig. 1. Patents describe the complete reduction of zinc and iron oxides approaches with focuses on producing a zinc-enriched fraction and an iron-enriched fraction (see Fig. 3). A literature survey and a patent survey revealed that hydrogen reduces iron oxide selectively and zinc is recovered in a subsequent process step. The

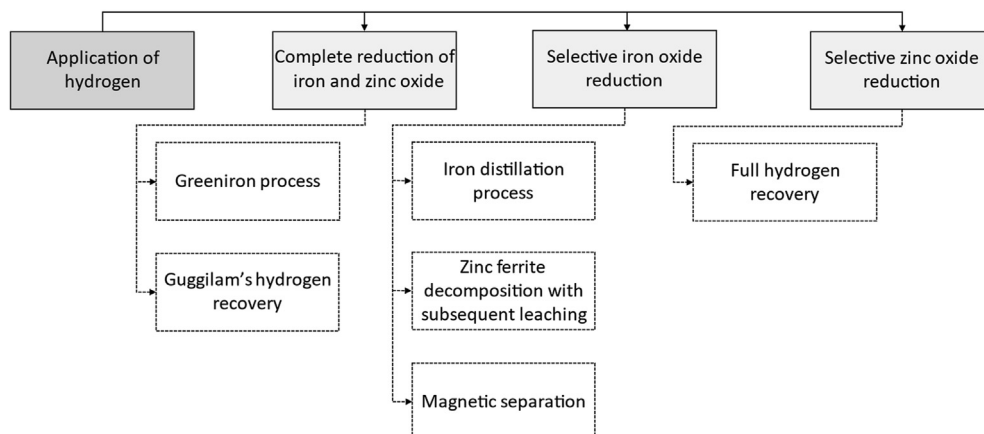


Fig. 2 – Process concepts using hydrogen as a reducing agent divided into three different categories. Complete reduction and selective iron oxide reduction are presented in literature and patent specifications [24–33]. Selective zinc oxide reduction is derived from thermodynamic calculations and is firstly proposed in this study.

selective zinc oxide reduction allows for full hydrogen recovery, which is a theoretically developed approach first proposed in this study (see Fig. 4).

Process concepts – current state of research

Complete reduction of iron and zinc oxide

The Greeniron process was initially developed to produce iron from iron oxide (see Fig. 3b) [24]. The raw material is treated batch-wise with hydrogen in a pressurized furnace with up to 10 bars. Electrical heating elements or indirect gas-fired radiation tubes provide the thermal energy to maintain a temperature of 650 °C. The generated water is continuously removed from the atmosphere via a condenser, continuously shifting the thermodynamic equilibrium towards reaction products. Nowadays, Greeniron H2 AB advertises the process as a solution to treat various types of industrial wastes,

including zinc-containing oxidic materials such as electric arc furnace dust. Zinc oxide is reduced to metallic zinc, which volatilizes in the reduction zone (higher temperature region). Continuous condensing of zinc in the condenser (lower-temperature-region) keeps its partial pressure low allowing high extraction rates for zinc to be achieved. The products are a zinc-enriched fraction captured in the condenser and a metalized iron-enriched fraction including inert oxides and non-volatile substances such as quartz, lime, and copper. The impure sponge iron is assumed to be recyclable within the electric arc furnace but must be kept under protective gas to prevent oxidation of the fine-grained material. In a similar concept, Gao et al. describe wet separation as a possibility to remove the inert gangue from the iron [25]. Washing with water can remove water-soluble halides, such as chlorides, from the zinc oxide, similar to the washing step for crude Waelz oxide [32,34].

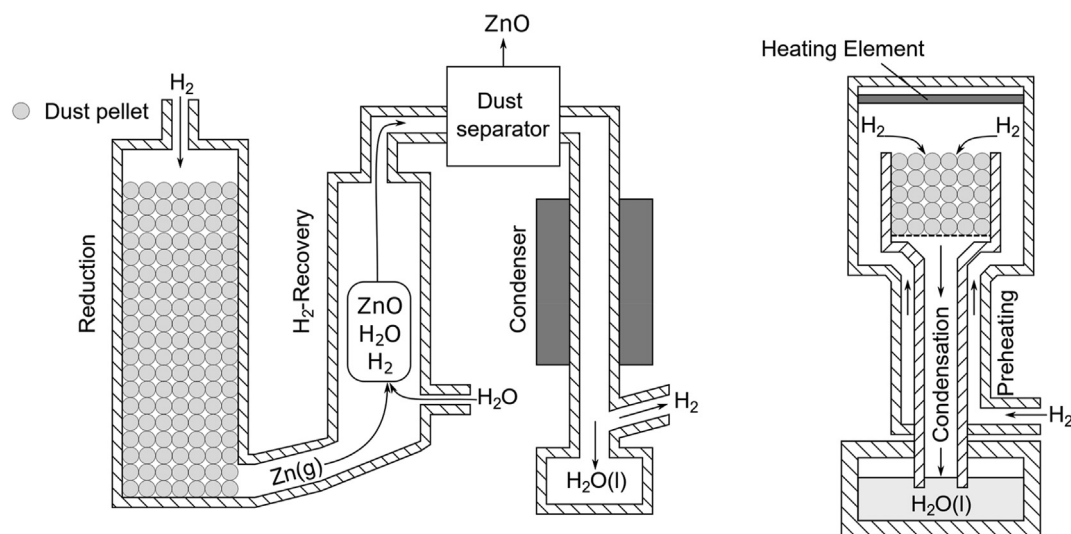


Fig. 3 – (a) Guggilam's concept for hydrogen recovery and (b) Greeniron concept redrawn and adapted from Refs. [24,26].

Guggilam et al. patented a recycling process for electric arc furnace dust in a batch-wise process that recovers a part of the reduction agent (see Fig. 3a). A hydrogen-rich gas flows through the reduction zone and reduces zinc and iron oxide at 1000–1100 °C. Zinc evaporates and leaves the reduction zone as part of the exhaust gas consisting of unreacted hydrogen and water vapor. The innovative character is the regeneration of the unreacted hydrogen by humidifying the off-gas to increase its oxidation potential. In this step, excess water injection enriches the gas with steam and cools it to range between 150 °C and 250 °C. The formed zinc oxide particles are separated in a dust separator, such as a cyclone or a baghouse filter. Condensation of excess water vapor enriches the hydrogen concentration to allow for recirculation back into the reduction zone. The product streams are comparable to those generated by the Greeniron process: A zinc-enriched dust fraction and metalized iron-oxide fraction. No information regarding product quality is available [26,27].

Selective iron oxide reduction

The iron distillation process is a two-step concept initially developed to produce zinc from iron-zinc concentrates [28]. In the first step a carbon monoxide atmosphere selectively reduces iron oxide to iron at 700 °C [29]. Fig. 1 indicates that hydrogen can be used as the reduction agent instead of carbon monoxide. A thermodynamic analysis from Pickles shows that increasing temperature shifts the thermodynamic equilibrium, which enables the metallothermic reduction of zinc oxide with iron at an elevated temperature [35]. Itoh et al. describe that the second step is ideally performed at 900–1200 °C in a vacuum or inert atmosphere. Gaseous zinc exits the reduction zone and condenses in a metallic form in a condenser [29]. The process was also evaluated for zinc-containing waste streams like flue dust from the steel industry [30]. The process generates a zinc alloy and an iron-oxide-enriched fraction containing inert oxides and more noble metals like Cu, which is recoverable via magnetic separation. The behavior of halides and other volatile compounds is not described.

Smith et al. proposed a selective hydrogen reduction of steelmaking flue dust as a possible pre-treatment for a hydrometallurgical process [31]. Hydrometallurgical approaches are feasible if zinc is leached selectively and iron remains in the solid residue to avoid the costly iron precipitation step. However, a significant amount of zinc in EAFD is bound as zinc ferrite from which selective leaching is not possible directly. The objective of the selective reduction step is the decomposition of zinc ferrite to zinc oxide and iron oxide to allow for a high zinc recovery rate without dissolution of iron in the subsequent leaching step. A similar concept was described by Antrekowitsch et al. [32]. Generated products and the behavior of accompanying elements can be found for various hydrometallurgical concepts in Refs. [36–40] with a detailed review written by Jha et al. in Ref. [41].

Itoh et al. proposed a treatment process in which EAFD is mixed with lime and heated to 900–1100 °C. The lime reacts with the zinc ferrite to zinc oxide and calcium ferrite that can be separated with the aid of a strong magnetic field producing an iron- and a zinc-enriched fraction [42]. The zinc-enriched fraction contains inert oxides and halides and has similar properties as Waelz oxide. The fine-grained iron-enriched fraction could be recyclable to the electric arc furnace. Considering [31], a similar outcome is possible by selective hydrogen reduction instead of lime addition to decompose zinc ferrite. A technique utilizing hydrogen reduction in combination with magnetic separation was described by Kruglick [33].

Novel process concept

Selective zinc oxide reduction

A concept derived from thermodynamic calculations and the literature survey in the present study is the selective reduction of zinc oxide combined with hydrogen recovery suggested by Guggilam et al. Area 3 in Fig. 1 illustrates the required process parameters in the reduction zone: elevated temperature and a low H_2-H_2O ratio. A higher H_2-H_2O ratio lowers the

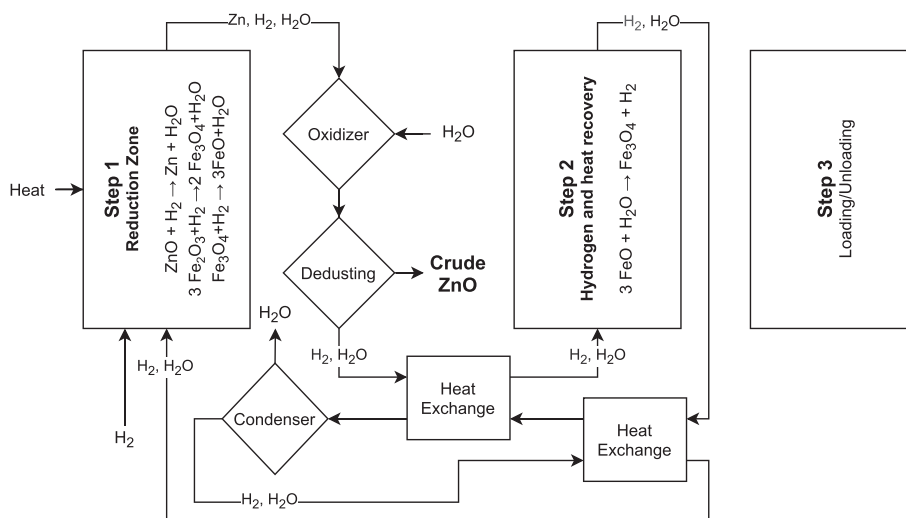
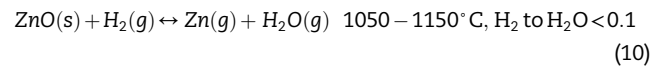


Fig. 4 – Flowsheet of novel process to distil zinc from electric arc furnace dust without consumption of reducing agent.

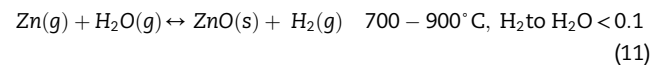
Table 1 – Comparison of the state-of-the-art concepts and a novel conceptualization for hydrogen-based EAFD recycling.

Process	Concept	Main product	Additional product	Temperature	Hydrogen consumption for reduction
Greeniron	Full reduction	Zinc condensate of unknown quality	Iron enriched fraction	650 °C	Highest
Guggilam's hydrogen recovery	Complete reduction	Zinc oxide enriched fraction similar to Waelz oxide	Fine-grained iron-enriched fraction	1000–1100 °C	High
Iron distillation	Two-step process	Zinc alloy of unknown quality	Iron oxide enriched fraction	1st step: 700 °C	High
Pre-treatment to leaching	1st step: selective iron reduction			2nd step: 900–1200 °C	
Pre-treatment to magnetic separation	Decomposition of zinc ferrite	Leachable zinc oxide	–	600 °C	Low
	Selective iron oxide reduction	Zinc oxide enriched fraction	Fine-grained iron	600 °C	Low
Suggested novel process	Selective zinc oxide reduction	Zinc oxide enriched fraction similar to Waelz oxide	Iron oxide enriched fraction	1050–1150 °C	Lowest

recirculation frequency of the gas, but also leads to the reduction of iron oxide to wustite and to the consumption of hydrogen. However, using multiple retorts allows recovering hydrogen from the unwanted iron reduction. Fig. 4 shows the process concept. Hydrogen primarily reduces zinc oxide to gaseous zinc, that exits the reduction zone together with the unreacted hydrogen and water vapor:



The significant advantage is that the process does not consume expensive hydrogen to produce an impure iron fraction. In the oxidizer, water is injected to cool the gas and react with zinc to zinc oxide and hydrogen:



At approximately 130 °C, a dust separator removes the particles from the gas. The resulting gas mainly consists of water vapor, which is preheated via a heat exchanger before it is used to reoxidize iron. In the heat and hydrogen recovery zone the steam-enriched gas oxidizes iron by generating hydrogen and heats up. The hot gas passes through heat exchangers before it enters the condenser that removes excess vapor until the H_2 to H_2O ratio reaches a value of around 2. A continuous batch operation requires a third retort for loading and unloading. An operation cycle consists of the following part steps: Reloading when the reduction is completed. Then, the reloaded retort goes into reduction operation and the reduced retort works as a recovery zone for hydrogen. The third retort can be unloaded and prepared for loading again.

Table 1 summarizes the presented processes ranked by the respective hydrogen consumption.

Conclusion

Hydrogen as a reduction agent in the scope of EAFD recycling enables innovative process concepts because it allows for a selective reduction of iron oxide, a selective reduction of zinc oxide and a complete reduction of both. Different conceptualizations partly implement these theoretically elaborated possibilities. Guggilam proposed a way to minimize hydrogen consumption by reoxidation of gaseous zinc to zinc oxide with steam. The Greeniron process operates at a lower temperature but at an increased pressure, which reduces the energy consumption by decreasing heat losses and lowering the required heating energy. The published information misses details regarding the condenser and the recyclability of the metallic iron fraction prone to reoxidation. The iron distillation process produces metallic zinc in a 2-step concept by distillation in a vacuum or protective gas atmosphere without the formation of oxidizing gases in the second step. This simplifies the condensation of gaseous zinc that is usually prone to reoxidation with decreasing temperature. However, the available publications miss data regarding accompanying metals and the behavior of halides. Smith et al. showed that a pre-treatment with hydrogen decomposes zinc ferrite to

increase the zinc recovery rates in a subsequent hydrometallurgical leaching process. According to thermodynamic considerations, Itoh et al.'s magnetic separation approach can also be applied after a selective iron oxide reduction. Thermodynamic considerations in combination with the conducted literature and patent study guided the authors to suggest a novel process with a minimized hydrogen consumption for reduction.

The variety of implemented and suggested concepts show a high potential to apply hydrogen as a reducing agent for EAFD recycling. A major advantage of hydrogen is the increased chemical kinetics allowing for a lowered process temperature, leading to fewer heat losses and lowering equipment wear.

These advantages are accompanied by drawbacks originating from an economic point of view, especially when compared to carbon-based technologies. Currently, hydrogen is more expensive than carbon. However, the innovative character of the described concepts shows that its application is possible with little hydrogen consumption. While the energy input for carbon-based reduction processes is often implemented by partial combustion of carbon, hydrogen-based concepts require external heating either electrically or with gas-fired tubes, increasing the operating costs. However, especially economic factors are likely to change in the future. The transformation towards renewable energy sources will increase the availability of hydrogen. In contrast to that, carbon dioxide taxes are likely to be implemented in many western countries. Additional research of high quality in this scope is necessary. This concerns the fundamental processability of the suggested process concept, the kinetical aspects and technological questions of the larger scale applications. Both circumstances – the change of economic conditions and the availability of high-quality research – will help to transform hydrogen into a viable reducing agent for green EAFD recycling concepts.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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