



# Recovery of Lead and Zinc from QSL- and IS-Slag using Hollow Electrode Carbon Insertion Process

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## 1 Introduction

During the transformation of primary raw materials to consumer goods the accumulation of residual substances cannot be avoided considering substantial economic and ecological efforts [15]. These substances have to be collected and transported to their final destinations which can be reprocessing or dumping. Primarily dumping is to be avoided, even though its quantity and injurious character has been decreased. As a consequence substantial and/or energetic utilisation of wastes has priority over their removal [1]. Dumping of residual substances is made more difficult by higher requirements in respect of environmental protection and steadily rising costs. Besides since the commencement of the Basel Convention for controlling the international transport of environmentally hazardous residual substances (1989), the reprocessing of residual substances has become of special interest [4].

Zinc containing residues such as slag from lead, zinc or copper industry, leaching residues or flue dusts, often contain pollutants like heavy metals or anions [15]. The European wide strict legislation concerning to dumping of heavy metal bearing substances will be intensified in the future. Main criteria for the decision between using or dumping these residual substances are the content of heavy metals and their water solubility. Thus the use of slags from Pb- or Zn-industry as construction material may become limited or even forbidden. Furthermore the high Zn and Pb contents in the used or deposited slag constitute metal losses which are unfavourable from the economic point of view. Because of metallurgical, economical or process engineering reasons heavy metal contents of less than 1 % Zn and 0.1 % Pb in the slag cannot be reached directly in primary Zn and Pb production. Subsequent slag treatment offers the possibility to recover the contained metals as well as to produce a slag product for the market. In a three years project sponsored by the Federal Ministry of Education and Research in cooperation of the RWTH Aachen University and three industry partners a procedure is developed to reach this value using a subsequent slag treatment in an electric arc furnace with hollow electrode technique.



## 2 Slags from pyrometallurgical zinc and lead production

World-wide 5.9 millions tons of lead are produced annually. Approx. 75% of it are used for the production of accumulators. Because of the low water solubility of lead, a hydrometallurgical winning cannot be applied economically, so that primary lead production takes place in the pyrometallurgical way nearly without exception.

World zinc production amounts to 7.2 million tons per year. Approximately 50% of the produced zinc quantity are used for corrosion protection [21]. Zinc is produced in hydrometallurgical processes followed by electrowinning for over 80%. The only pyrometallurgical procedure for zinc winning which plays a substantial role nowadays, is the Imperial Smelting process which also serves for lead production [9].

### 2.1 Slags from thermal zinc production

Pyrometallurgical zinc takes place in the Imperial Smelting furnace (IS Furnace). In the past, solely sinter from sulphidic concentrates were treated. Meanwhile the aggregate is used intensively for the processing of residual substances, which leads to a reduction of sulphureous materials in the load. Nevertheless a significant sulphur concentration is measurable in the slag.

The IS process is granulating slag in a water spray. The high Zn-content of the slag represents a zinc loss that cannot not be avoided. A further Zn-reduction would be accompanied by an iron reduction, which leads to solid iron in the shaft and to a damage of the refractory lining. An increase of the process temperature to keep the iron liquid would result in an intensified energy consumption, an additional liquid phase (e.g. pig iron) and an increase of lead evaporation.

### 2.2 Slag from the QSL process

The Queneau Schuhmann Lurgi process combines roasting and reduction reactions in one reactor, utilizing the roasting energy for smelting and reduction [14]. Because the oxidation does not proceed completely, a PbO rich slag is formed beside the crude lead (98 % Pb). This slag is reduced in the reduction zone by coal injection [9, 18]. Here most of the charged zinc is collected in the slag. Only a minor part is withdrawn as Zn-gas or ZnO in the exhaust gas. The final slag possesses also a high lead content.

### 3 Proved procedures for the treatment of lead and zinc slags

#### 3.1 Slag fuming

Slag fuming is a volatilisation process, where zinc, lead and tin from a liquid slag are transferred into a oxidic flue dust [3, 16]. Fig. 1 shows schematically the principle procedure of the slag fuming.

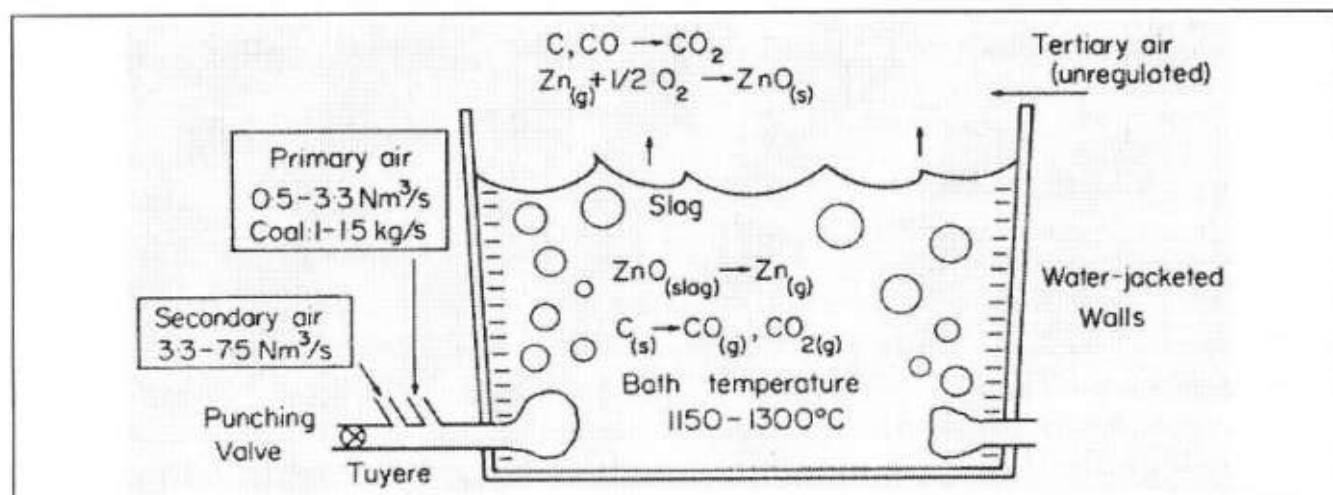


Figure 1: Schematic diagram of the slag fuming procedure [19]

Fine grained coal (< 0.1 mm) is injected into the slag bath by air (so-called primary air) [24]. Together combustion air (secondary air) that corresponds to approx. 60-75 % of the theoretical air requirement is injected [7]. At the beginning of the process as well as before tapping the air/coal ratio is increased briefly in order to increase the bath temperature by coal combustion. Coal serves as fuel and reducing agent. It can also be replaced by oil and/or natural gas [12, 19]. The metal oxide reduction takes place at 1150 - 1300°C according to following equations:



Above the slag bath the metal vapour oxidizes with tertiary air, and post combustion of the gas takes place:



Subsequently, the gas is cooled and the dust is collected in filters and/or dust precipitators. The use of the offgas heat (e.g. for power generation, preheating of injected air) is decisive for the economical efficiency of the process [7]. Slag fuming is mainly a batch process; continuously working systems exist, however they play a minor role [19]. The liquid slag is charged into the water-cooled reactor, where also solid residues such as slag granulates, can be charged [9, 24]. Usual reduction times vary between 50 and 100 minutes, but they can reach up to three hours [3, 22, 23]. The final

Zn content amounts typically between 1.5 and 3.3 %, With very long reduction times values below 1 % in final slag can be reached [5, 23]. The Pb-content at the end of the slag fuming amounts to 0.03 - 0.05 % [7, 24].

Disadvantages of slag fuming are the large quantities of exhaust gas and the complexity of the offgas treatment. The operation of a slag fuming plant is only feasible if an appropriate steam consumption is given [9]. Another disadvantage is the high demand for coal due to its double application as fuel and reducing agent. Besides it is impossible to recover the zinc in metallic form because of the high oxygen potential in the exhaust gas. So the produced zinc oxide has to be reduced again in the next step.

### 3.2 Plasma process

A modification of slag fuming process is the Plasmazinc process developed by SKF Steel engineering [6]. With this procedure the energy for the highly endothermic reduction reactions is delivered by plasma. Due the fact that the plasma is inert, a coke bed, which fills the reactor, serves as reducing agent. The crude material is charged into the reduction shaft by cone. Fines are injected into the plasma steam. The plasma and the gases that appear during reaction form a cavity in front of each blastpipe. The reduction reactions mainly take place in a 100 mm thick coke bowl around the cavity.

The Plasmazinc process works with a surplus of coke, so the exhaust gas is rich in  $H_2$  and CO and poor in  $H_2O$  and  $CO_2$ . Compared to the slag fuming procedure, the Plasmazinc offgas possesses a lower oxygen potential. Thus the zinc can be regained in metallic form. Informations about obtainable Pb and/or Zn-contents in the tapped slag are not published. Generally spoken Plasma processes are suitable for applications, which need high temperatures and strong reducing conditions. However in large scale their complexity and their high costs (e.g. coke) have an unfavourable effect [11].

### 3.3 Ausmelt or Isasmelt process

The Ausmelt or Isasmelt process is also suitable for the removal of lead and zinc from slags. Like in case of slag fuming, coal as reducing agent and solid fuel is injected through a lance [17]. The final slag contains < 0.1% Pb and < 1.0% Zn and can be used e.g. for road construction [11]. This process differs from the slag fuming procedure only in the equipment design and has accordingly the same disadvantages: large quantities of exhaust gas, a complex offgas system, high demand for coal and reclamation of the zinc in oxidic form.



## 4 Thermodynamics of zinc reduction

The following equation applies generally for the reaction between zinc and oxygen:



The oxygen potential at the thermodynamic equilibrium is represented by the following equation

$$RT \ln p_{\text{O}_2} = \Delta H^\circ - T \left( \Delta S^\circ - R \ln \frac{a_{\text{ZnO}}^2}{a_{\text{Zn}}^2} \right) \quad (6)$$

with  $\Delta H^\circ$ : standard enthalpy (J);  $\Delta S^\circ$ : standard entropy (J/K); T: temperature (K); R: gas constant (8.314 J/K); K: equilibrium constant;  $a_i$ : activity of the material i;  $p_{\text{O}_2}$ :  $\text{O}_2$  partial pressure (Pa)

However this formula does not mirror the real reaction procedures. The influence of the activities is neglected. In improvement of the thermodynamic calculation for Zn and Pb-reduction two slag compositions are considered: industrial slag and a specific final slag with 0.1 % Pb and 1.0 % Zn. The activity is linked with the mole fraction by a coefficient:

$$a_i = \gamma_i x_i \quad (7)$$

with  $a_i$ : activity of the element;  $\gamma_i$ : coefficient of activity of the element i;  $x_i$ : mole fraction of the element i

The activity coefficient of solid zinc oxide was determined equally. According to Battle and Hager [2] between 1100°C and 1300°C following equations:

$$\begin{aligned} \gamma_{\text{ZnO}} = & 6,39 x_{\text{Al}_2\text{O}_3} + 11,8 x_{\text{CaO}} + 7,32 x_{\text{FeO}} \\ & + 3,74 x_{\text{Fe}_2\text{O}_3} + 7,15 x_{\text{ZnO}} - 3,31 \end{aligned} \quad (8)$$

whereby the absence of  $x_{\text{MgO}}$  and  $x_{\text{SiO}_2}$  in the equation is to be attributed to a lack of data. Using exclusively this model temperatures lower than 1100°C and above 1300°C can not be considered. Furthermore it is a fact that activity coefficient depends by temperature. Battle and Hager's equation does not consider this detail.

From Kellogg [8] was found for the whole temperature range:

$$\gamma_{\text{ZnO}} = \exp \left( \frac{920}{T} \right) \quad (9)$$

This calculation leaves interaction effects between the slag components unconsidered. By considering interactions between the components the activity coefficient will decrease. If both investigations are matched to a modified activity coefficient as follows. The data are related to the average temperature (1200°C) of Battle and Hager's investigation:

$$\gamma_{\text{ZnO}} = \gamma_{\text{ZnO}}(1200^\circ\text{C}, \text{Battle}) \exp\left(\frac{920}{T} - \frac{920}{1473}\right) \quad (10)$$

Fig. 2 shows the ZnO modified activity coefficient as a function of temperature.

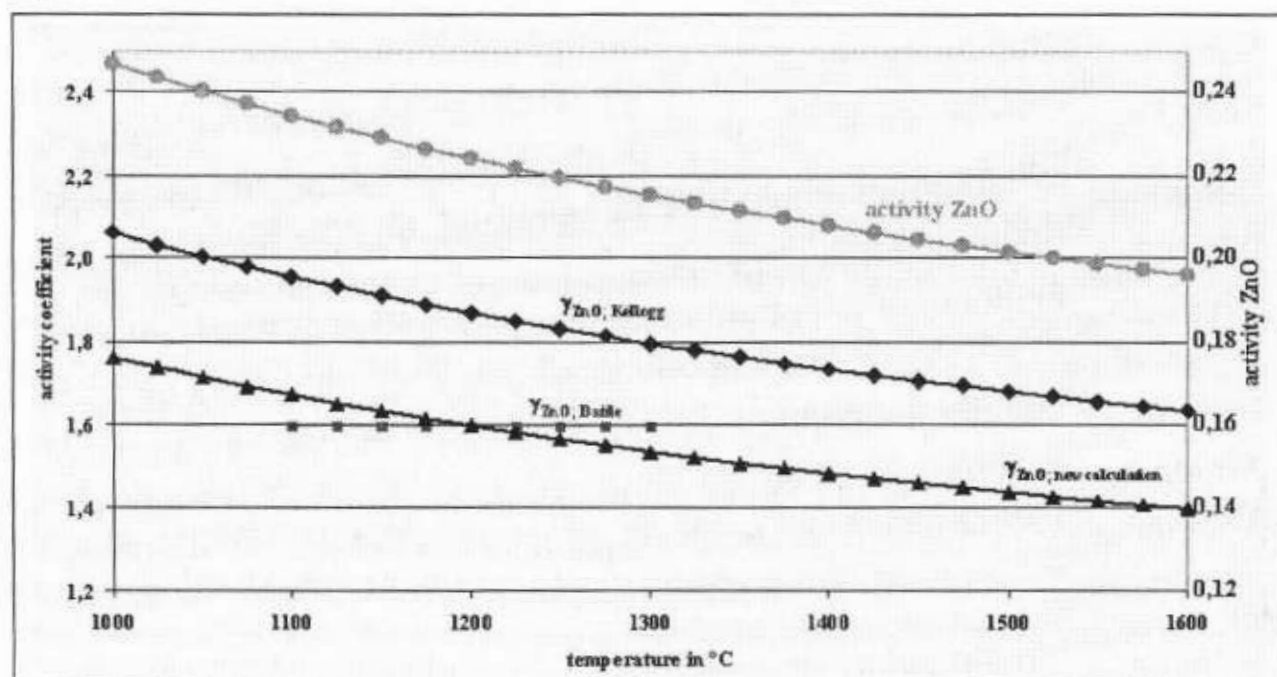


Figure 2: Modelled ZnO activity coefficient in QSL slag;

$$x_{\text{ZnO}} = 0,140; x_{\text{Al}_2\text{O}_3} = 0,026; x_{\text{CaO}} = 0,125; x_{\text{FeO}} = 0,295; x_{\text{Fe}_2\text{O}_3} = 0,010$$

Similar models are created for the activity coefficients of FeO, Fe<sub>2</sub>O<sub>3</sub> and PbO. Fig. 3 shows the modified oxygen potentials as a function of temperature for QSL-slag. Thermodynamically considered, slag temperature must be above 1200°C to promote the reduction of ZnO with carbon and to avoid a FeO reduction.



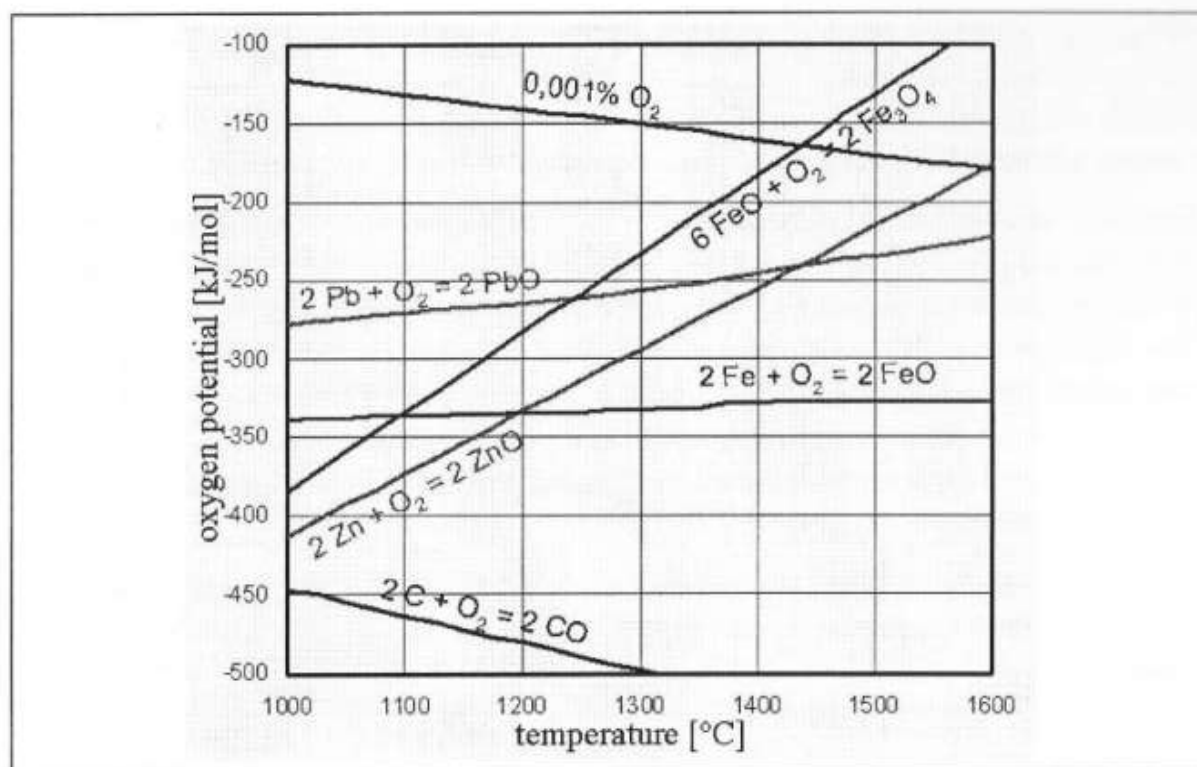


Figure 3: Oxygen potentials in the slag as functions of the temperature of OSL-slag;  
 $x_{\text{ZnO}} = 0,140$ ;  $x_{\text{Al}_2\text{O}_3} = 0,026$ ;  $x_{\text{CaO}} = 0,125$ ;  $x_{\text{FeO}} = 0,295$ ;  $x_{\text{Fe}_2\text{O}_3} = 0,010$

## 5 Kinetics of zinc reduction

The kinetics of zinc reduction were particularly examined for the slag fuming process. About the reaction mechanism and the speed-determining reaction step different contradictory theories prevail, which are summarized here.

According to Bell, Turner and Peters the large surface and the strong bath circulation during slag fuming lead to a very fast material and heat exchange, so that the thermodynamic equilibrium (equation 5) between slag and gas is always reached [16, 23]. The Zn-evaporation is directly related with the carbon supply [8]. With a constant addition of coal the Zn-content in the slag decreases linearly, which was actually observed [19, 24]. But comparing different slag fuming plants, no relationship between the  $\text{CO}_2/\text{CO}$ -ratio in injected air and the Zn-evaporation rate was found [23].

According to Quarm the Zn-reduction is a first order reaction concerning to the ZnO content [19]. The reaction rate is shown by the following equation:

$$\frac{d}{dt} c_{\text{ZnO}} = -k_1 c_{\text{ZnO}}(t) \quad (11)$$

with  $k_1$ : reaction rate constant ( $\text{min}^{-1}$ );  $c_{\text{ZnO}}(t)$ : ZnO concentration in the slag at time  $t$  (wt.-%);  $t$ : time (min)

By integration of this equation the degression of the ZnO cocentration can be computed:

$$c_{\text{ZnO}}(t) = c_{\text{ZnO},t=0} \exp(-k_1 \cdot t) \quad (12)$$

However a linear reduction of the ZnO content was observed in production.

Kinetics of the Zn-reduction can be discribed by two criteria. Macroscopically the penetration depth of the coal particles into the slag and their even distribution play a crucial role in the slag bath. Within the microscopic range the Zn-Reduction rate is first determined by the Boudouard reaction. The formation of a CO bubble around an injected coke particle is a substantial procedure. Subsequently the material transfer in the slag becomes the speed-determining step. The Zn-gas pressure as a mechanism for the Zn-evaporation is represented in fig. 4. It becomes more and more crucial at higher Zn-concentration. A turbulent circulation of the electrolyte and a high temperature are accelerating this effect.

For zinc reduction by slag treatment a high bath turbulence is essential. This is realised by an intensive gas flow gas in slag fuming process. Electric arc furnaces also realise extreme mass transfer.

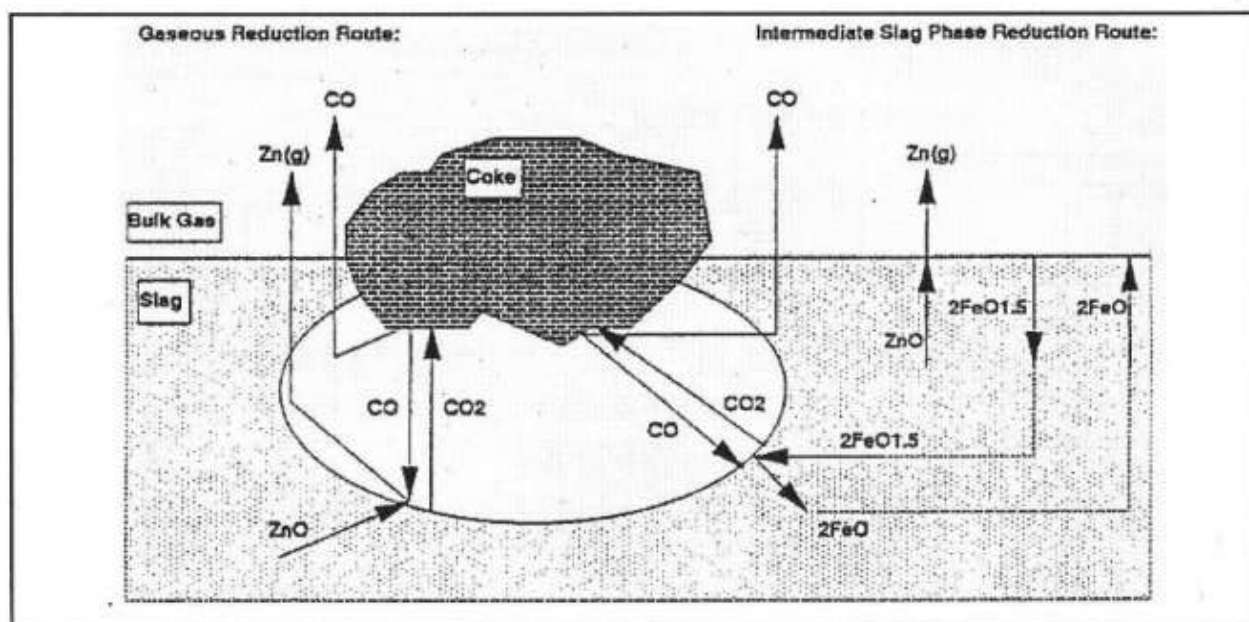


Figure 4: Reaction mechanisms of the Zn-reduction in liquid slag [20]

## 6 Hollow electrode technology for EAF processing

The most essential point of this investigation is the injection of carbon as reducing agent into a liquid slag without using reaction gas. Only nitrogen is used to avoid a flow of slag in the electrode cavity. Coke particles shall penetrate into the slag bath as deep as possible in order to maximize the retention time of this particles in the slag. The nitrogen flow through a hollow electrode into the slag is examined by means of a water model on a scale of 1:1. In this model the furnace is replaced





by a transparent vessel made of plastic. Compressed air, whose flow rate and pressure are variable, simulates the nitrogen supply during the process. Water is used in the model to simulate a liquid slag. In order to transfer the results of the water model to the arc furnace, dimensionless similarity indicators must be approved. The similarity indicator relevant for the movement of gas bubbles is the densimetric Froude number, specified by the following equation [13]:

$$Fr' = \frac{u_g^2 \cdot \rho_g}{g \cdot \rho_l \cdot l} \quad (13)$$

with  $u_g$ : gas speed (m/s);  $\rho_g$ : gas density (kg/m<sup>3</sup>);  $g$ : acceleration due to gravity (9.81 m/s<sup>2</sup>);  $\rho_l$ : liquid density (kg/m<sup>3</sup>);  $l$ : characteristic length (m)

To adjust the  $Fr'$  similarity the following equation must apply:

$$\frac{u_{N_2}^2 \cdot \rho_{N_2}}{\rho_s} = \frac{u_A^2 \rho_A}{\rho_w} \Rightarrow u_A = u_{N_2} \sqrt{\frac{\rho_{N_2} \cdot \rho_w}{\rho_A \cdot \rho_s}} \quad (14)$$

with  $N_2$ : Nitrogen; A: air; S: liquid slag; W: water;  $\rho_{N_2}$  (20°C) = 1,165 kg/m<sup>3</sup>;  $\rho_s$  (1200°C) = 1,165 kg/m<sup>3</sup>;  $\rho_w$  (20°C) = 998,4 kg/m<sup>3</sup>;  $\rho_A$  (20°C) = 1,188 kg/m<sup>3</sup>;  $u_{N_2}$  (max.) = 0,236 m/s = 10 l/min

The result is a gas velocity of 0.120 m/s for the compressed air in the model, which corresponds to a flow rate of 5.0 l/min. Fig. 5 shows the bubble evolution in the water model with adjusted  $Fr'$  similarity. The bubbles formed at the bottom of the hollow electrode do not penetrate deeply into water but rise immediately to the surface. It can be assumed that the  $N_2$ -bubbles in behaves equally in the slag bath. The investigations in the water model show that an effective insertion of coke particles into a liquid slag can be accomplished. The gas does not play a significant role to the penetration of the particles into the slag.

The injecting system of the furnace is extended by a wheel air-lock cell in order to reach appropriate gas tightness controlled. The wheel air-lock permits a slow and adjustable charge of coke into the arc furnace, which leads to a controlled reaction between slag and coke. Coke is charged by a coke column above the wheel air-lock. Wheel air-lock and coke column ensure a sufficient sealing of the upper part.

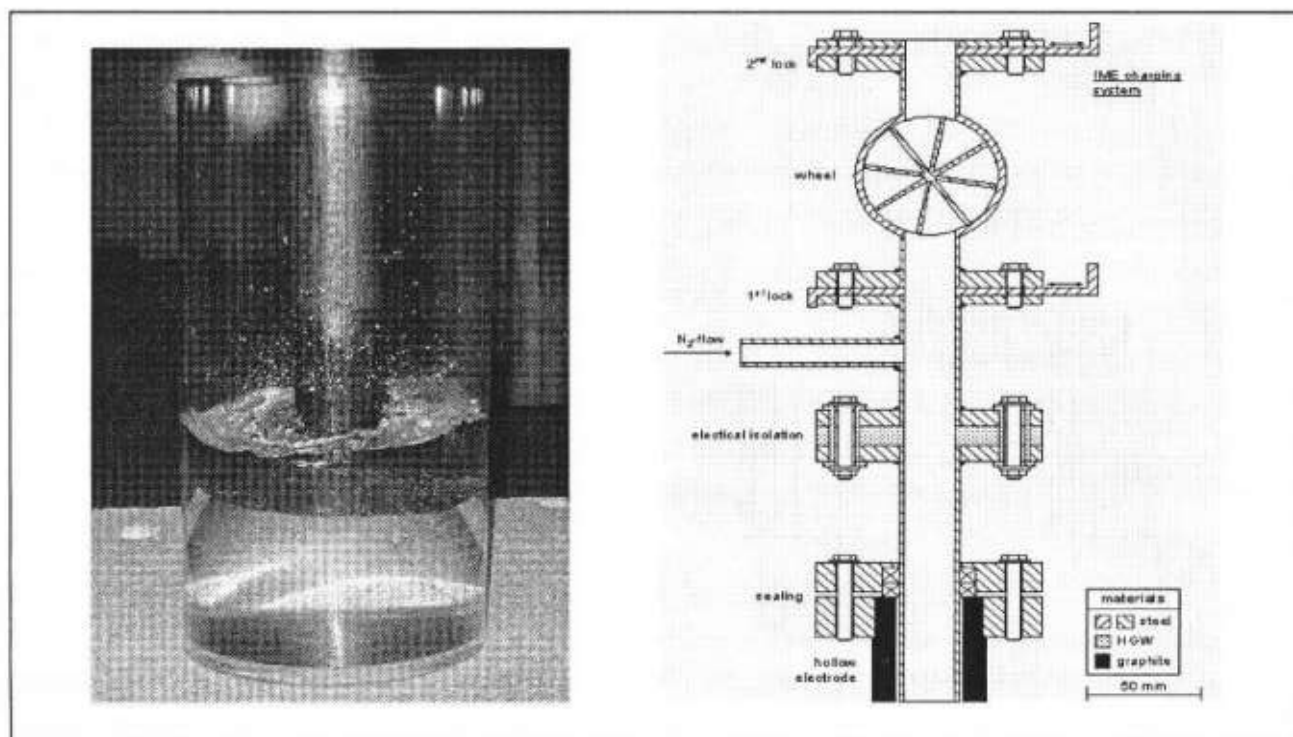


Figure 5: Water model of hollow electrode    Figure 6: For laboratory EAF developed charging system for coke by hollow electrode

## 7 Zinc reduction by hollow electrode system in laboratory scale electric arc furnace

IME has an laboratory arc furnace with a capacity of approximately 6 l. The refractory lining consists of MgO and is surrounded by an 8 mm strong steel shroud. This furnace is provided by a water cooled bottom anode of copper. The graphite cathode is adjustable by a hydraulic system. Another hydraulic enables the furnace to be tilted for tapping/casting. The electric current supply of the furnace is secured by a 52 kVA electric rectifier. An exhaust ring seizes the offgas and leads it to a water cooled spark separator and cloth filter, in which the flue dust is separated from the gas. As fig. 7 shows the inlet and outlet temperatures of the cooling water, the temperature of the furnace feeds as well as the temperatures on different positions of the bottom electrode and the waste gas system are continuously measured.

On the other hand the measurement of the melt temperature takes place by a thermocouple lance discontinuously.

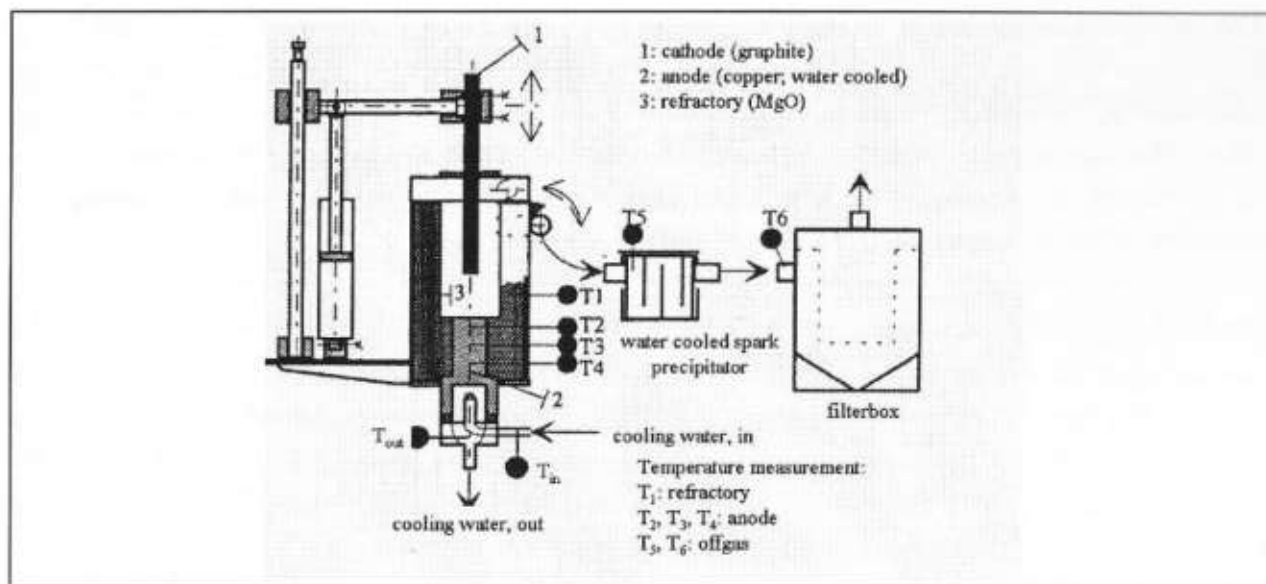


Figure 7: Schematic structure of the laboratory arc furnace

The first preliminary tests in the laboratory arc furnace are to examine the meltability and reduction behavior of the slag, before the developed charging system is to be used in the current project.

Excluding slag from the QSL-process with a content of 12.7 % Zn and 9.6 % Pb was used for this investigation.

The charging of lignite took place via the hollow electrode. As tapping temperature approx. 1400°C is aimed. After solidification of the slag in a mold, the slag block is removed and minced to be analyzed. The flue dust samples are taken from the separator. During the process slag samples are taken from the furnace by a lance. The final samples consist of material that are granulated during tapping.

## 7.1 Statistic investigation of the results

A statistic analysis of these results is accomplished, in order to determine the influence of each parameter on the Pb and/or Zn-reduction.

The regression procedure is described considering as example the Pb-content in the fine fraction (< 90 µm) %Pb<sub>f</sub>. At first the different Pb-content in the final slag are compared with one another in order to determine the most important research parameter for Pb-reduction. The importance of a parameter is given by the correlation coefficient  $R^2$ :

$$R^2 = \frac{\left( n \sum_{i=1}^n x_i y_i - \sum_{i=1}^n x_i \sum_{i=1}^n y_i \right)^2}{\left[ n \sum_{i=1}^n x_i^2 - \left( \sum_{i=1}^n x_i \right)^2 \right] \left[ n \sum_{i=1}^n y_i^2 - \left( \sum_{i=1}^n y_i \right)^2 \right]} \quad (15)$$

n: number of values;  $x_i$ : value of a parameter with in test run which can be examined;  $y_i$ : value of a target parameter in test run



The correlation coefficient  $R^2$  is one if all pairs  $(x_i, y_i)$  lie on the same straight line and is zero, if the parameter  $x$  does not have any influence on the target parameter  $y$  [10]. Fig. 8 gives the correlation coefficient  $R^2$  between different parameters and the lead content in the fine fraction and/or its logarithm. Thus as parameter with the highest correlation coefficient, and so to say the largest influence on the results, the reduction time  $t_R$  is determined. Fig. 9 shows the clear interrelation between the logarithm of the Pb-content in the final slag and the reduction time.

	<b>log (%Pb<sub>f</sub>)</b>
Cokes feed $m_K/m_S$	0.154
Cokes grain size $d_K$	0.048
Supplied Nitrogen volume $V_{N_2}$	0.259
Average electric performance m.e.L.	0.052
Casting temperatur $T$	0.006
Reduction time $t_R$	0.715
Total test run time $t_N + t_R$	0.443
Slag basicity $\%CaO/\%SiO_2$	0.050

Figure 8: Correlation coefficient  $R^2$  between the Pb-content in the fine fraction %Pb<sub>f</sub> and different parameters

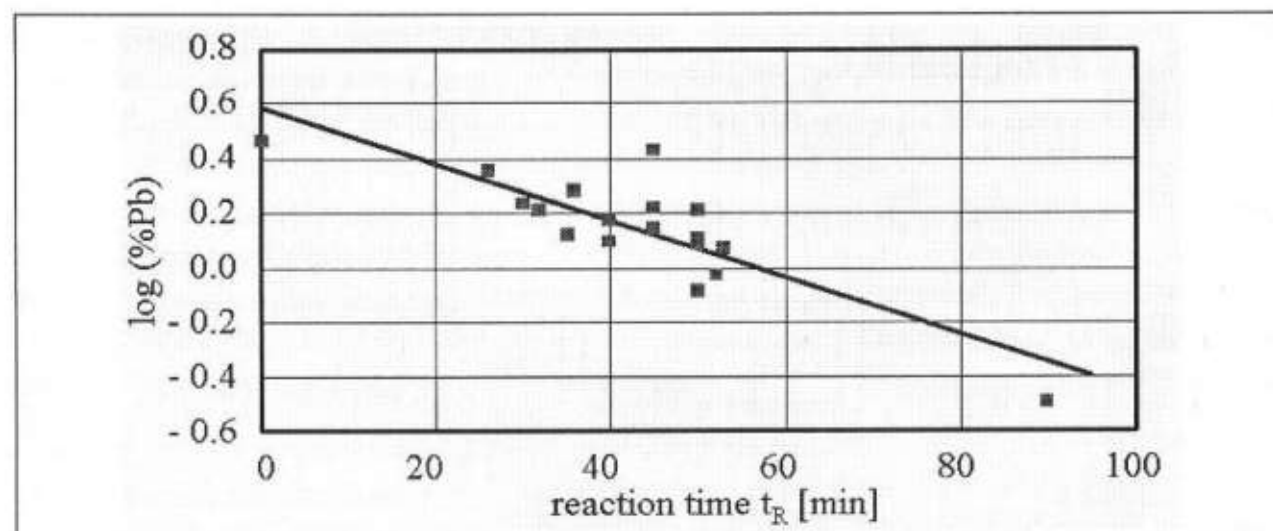


Figure 9: Pb-concentration in the QSL slag as function of the reduction time

A linear regression between  $\log (\%Pb_f)$  and  $t_R$  can be carried out (fig. 9). On the basis of these values for each test run a theoretical Pb-content can be calculated that only considers the influence of the reduction time  $t_R$ :

$$c_{Pb_f}(t_R) = 10^{m t_R + b} \quad (16)$$

$m$  = Gradient of regression line;  $b$  =  $\log$  (start concentration)

This theoretical Pb-content is subtracted afterwards from each corresponding Pb-content, so that the influence of the reduction time is abolished. The remaining Pb-content  $\%Pb_f - \%Pb_f(t_R)$  is compared with the remaining parameters on the basis of the correlation coefficient again. However in the further process the  $R^2$ -value does not permit any conclusion concerning to further factors with above-average influences. It can be concluded that the lead content in the final slag is determined by the reduction time. The parameter that plays the largest role in Zn-reduction is the MgO content of the slag ( $R^2 = 0.827$ ). Fig. 10 shows clearly the correlation between Zn and MgO content.

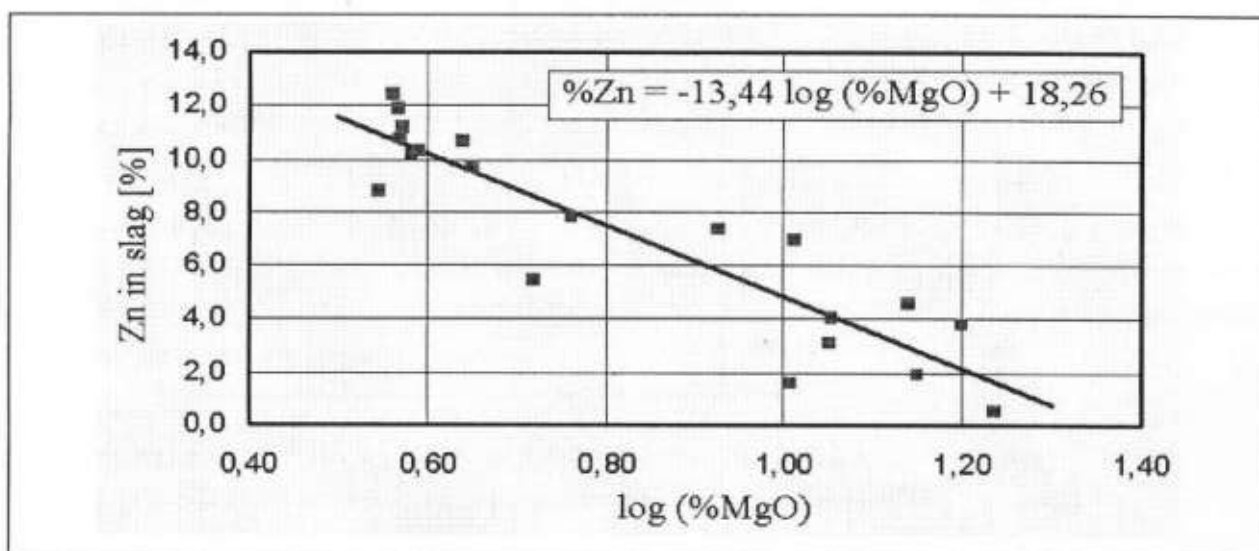


Figure 10: Zn-content  $\%Zn$  as function of the logarithm of the MgO content

The MgO content in QSL slag is increased by addition of MgO or by consumption of the refractory lining. The influence of MgO on the Zn-reduction is probably based on the increase of the ZnO activity.

Another parameter that exhibits a high correlation coefficient is the logarithm of the product of power consumption and total test run time  $t_N + t_R$ , that represents the electricity supply to the process ( $R^2 = 0.650$ ). In contrast to this no correlation between the remaining Zn-content  $\%Zn - \%Zn(\%MgO)$ , which is raised by the influence of the MgO, and this parameter can be observed. It can be concluded that the supplied energy does not influence directly the Zn-reduction. The low Zn-content with high power consumption and long test run duration is caused by the intensified dissolving of the MgO lining.



## 7.2 Kinetics of Zn-reduction in slag

The sampling took place via solidification of slag at granulation. Fig. 11 shows the graph of the Zn-content during a reduction experiment with QSL slag. After the first coke addition an almost linear reduction of the Zn-content can be observed, what coincides with the above mentioned theory. Only after 120 minutes the Zn-reduction is slowed down. Between the last sampling at  $t = 120$  min and tapping at  $t = 135$  min the Zn-content decreases from 0.69% to 0.56%. Thermodynamically the lower Zn-reduction ratio at the end of the experiment can be traced back to the reduced ZnO activity. From the kinetic point of view the slowing down of the Zn-reduction can be explained by the less favourable material transfer of the very low concentrated  $\text{Zn}^{2+}$ -ions to the reaction place. Both effects will superimpose each other.

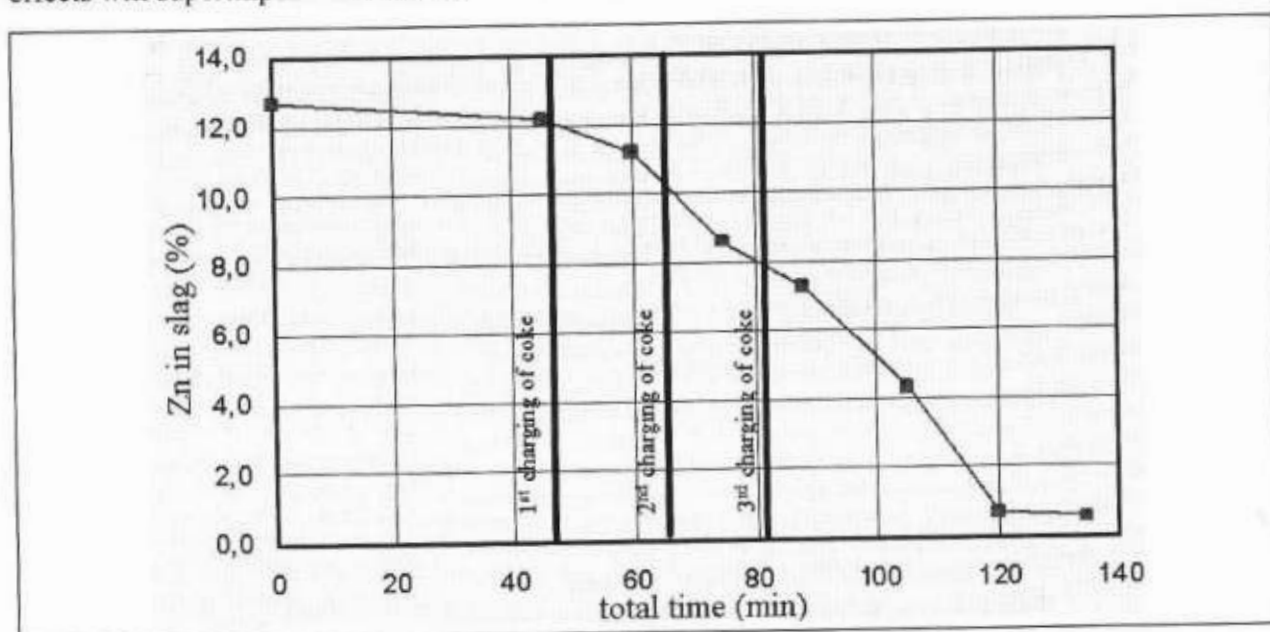


Figure 11: Zn-content in QSL slag as function of time

The investigation of the Zn-Reduction kinetic shows that very low Zn-contents (below 1.0% Zn) are attainable in the final slag under the following conditions:

- Long reaction times
- Large coke feed



## 8 Summary

Based on investigations of Kellogg and Battle/Hager modified activity coefficient of ZnO as a function of temperature was found. This allows the adoption for "real" slag systems. Thermodynamic and kinetic considerations suggest the requirement of process temperature above 1200°C and high slag flow for realising an effective mass transfer.

The investigations pointed out that the combination of arc furnace processing and charging of carbon bearing material by hollow electrode is a favorable combination in order to reduce zinc and lead contents in slags and supply the metals to the material cycle.

The development of a new technology for slag treatment is pushed by a restrictive legislation, that demands low heavy metal contents in residual substances. The charging of coke and the abandonment of gas insertion make this possible avoiding large offgas volumes. In the arc furnace also the temperature that is necessary for the process can be adjusted without any problems. A reduction of iron can only partly be inhibited; but local formation of metallic iron is not problematic since solidified iron will easily be remelted by the electric arc. The formed metallic iron will be available as reducing agent for further ZnO reduction.

In these slags lead is usually present in a metallic form. The lead reduction of the oxidic portion of the slag that originates from the QSL process depends on the reduction time. The electrical power of the arc furnace, the quantity of charged coke and the addition of fluxes do not exert considerable influence on the Pb-reduction.

The results show that the MgO content of the slag exerts a large influence on the Zn-reduction in QSL slag besides reaction time. The Zn-evaporation is favoured also by increasing electrical power, which represents a larger energy consumption. Kinetic influences prevail only with low zinc contents, because the EAF provides for a good circulation of the electrolyte.

Future work will check the influence of the sulphur content. Test runs in pilot scale are up to be conducted late 2003.

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