Metallurgical fundamentals for an autothermal melting of WEEE in a top blown rotary converter

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Abstract

The recycle flow of used electric and electronic devices is rapidly increasing in Western Europe because of the WEEE-directive 2002/96/EC from 27.01.2003. In 2006 about 7 Mio t of WEEE (**W**aste of **E**lectronic and **E**lectric **E**quipment) were collected within the EU-25. But only 50 % of that amount is recycled in these countries. Containing metals like copper, silver, gold, selenium, tellurium, indium, nickel, tin and lead build up a value between 1 000 and 4 000 €/t_{scrap} depending on the commodity price. Despite this economic and ecological point of view only 50 % of WEEE is recycled in Europe. Besides the high content of valuable metals WEEE also contains plastics, ceramics and glass, which contain elements like Bromine, Chlorine and Fluorine causing special attention.

IME and Aurubis are currently investigating a new process based on a top blown rotary converter (TBRC) aiming for an autothermal recycling of electronic scrap. Based on thermochemical modelling results a suitable slag for the process was validated in lab trials. Further experiments investigated the influence of the preconditioning on the metallurgy as well as oxygen consumption. The results allow for predicting a process window for the recycling process in the TBRC. First recycling trials in a 0,6 m³ demo scale TBRC with preconditioned material are planned and described. Finally an outlook will be given on process scalability and energy balance.

Introduction

The WEEE directive 2002/96/EG from 27.01.2003 of the European Union defines "**E**lectric and **E**lectronic **E**quipment (EEE)" as devices, which are dependent on electric currents or electromag-

netic fields in order to work properly or which is an equipment for the generation, transfer and measurement of such currents and fields up to an AC-voltage of 1000 volt or a DC-voltage of 1500 volt.

An estimate of the WEEE arising across the EU27 is between 8.3 and 9.1 million tonnes per year for 2005. This amount is about 4 % of the municipal waste stream. A number of forecasting assumptions were applied which predict that the total WEEE arising will grow annually between 2.5 % and 2.7 % reaching about 12.3 million tonnes in 2020. [1]

In 2008 across the EU27 10.3 million tonnes of EEE is put on market. But only 3.2 million tonnes of WEEE were collected in the same time. [4] This difference leads to the assumption that a lot of WEEE is exported illegally into countries outside EU27.

Electronic scrap is a complex mixture of a lot of different materials. It consists of about 15 to 30 % plastics, 40 to 50 % ceramics and 20 to 30 % metals like copper, aluminium, iron etc. Fig. 1 presents more details about an average composition of a common WEEE mixture. The composition depends on the age, origin and manufacturer of the EEE. [2], [3]
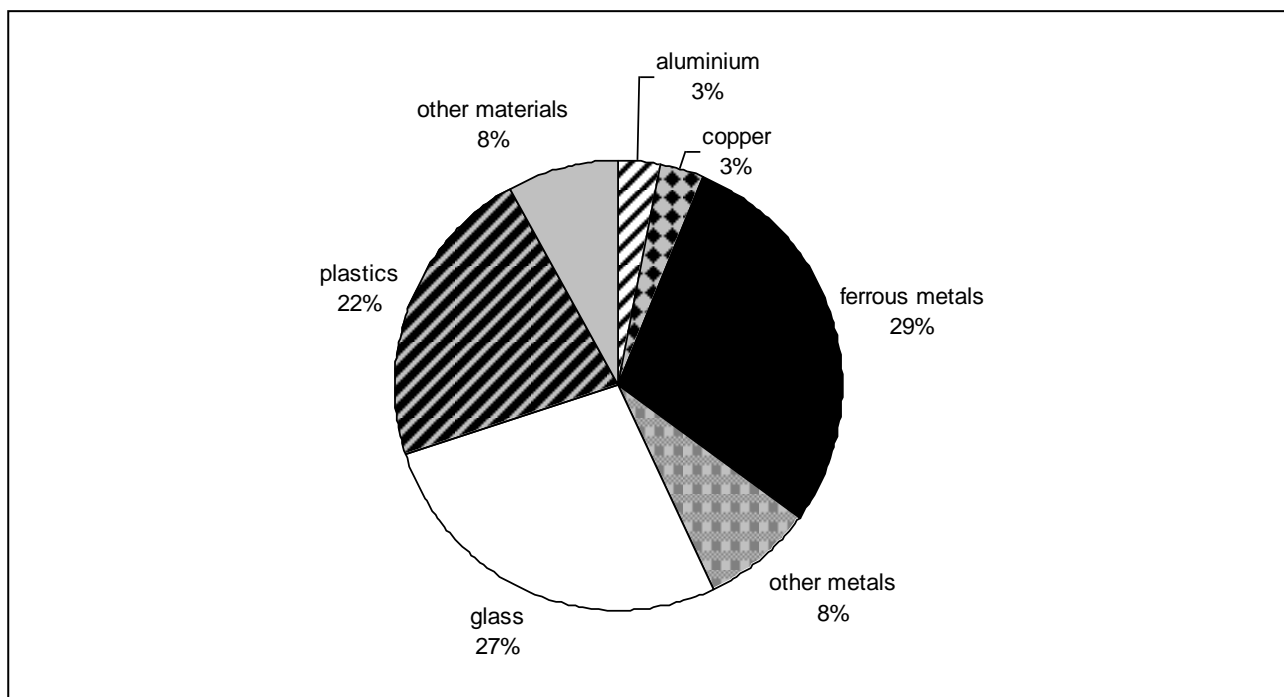

Figure 1:   average composition of WEEE [5]

A non-ferrous metal fraction can be generated by dismantling, crushing, iron and metal separation, grinding, milling and sorting. But there is a lot of carry over due to an imperfect liberation and separation of the highly complex composites.

There are four strong forces that motivates for recycling of WEEE. One force is protecting the environment as a lot of hazardous materials like lead, halides or arsenic can be found in WEEE-scrap. In the case of landfill deposition, hazardous materials are solubilised by rainwater and may pollute the environment. Another force is of economical nature. High valuable metals such as copper, silver,

gold, platinum and palladium are contained in WEEE. After preconditioning the commercial relevance ranges between 1 000 and 4 000 €/t_{scrap} for the copper rich fraction depending on the commodity price. The high value metals together with Cobalt, Indium, Antimony, Tantalum, Selenium and Tellurium are strategic metals in the EU because they are necessary to keep our high technological standards and safeguard our future developments [7]. Such keeping these materials inside the borders of the EU is also a social force through connected working places. The implementation of the WEEE directive 2002/96/EG from 27.01.2003 in the EU member states is a legal force. Up to the year 2016 a recycling quote of 65% will come into force.

In this article basic investigations of the melting behaviour after different pretreatment processes like pyrolysis and combustion are described with an outlook to first large scale tests in a TBRC at the IME currently conducted.

Combustion and pyrolysis of WEEE

As part of the development for a new innovative process minimizing emissions the origin of all materials has to be known. The high variety of organic compounds in electronic scrap makes this investigation complex. In the past several investigations ([9] to [14]) deal with the formation of dangerous materials like furans and dioxins. All investigations also had shown the presence of bromine. The pretreatment of materials for metallurgical processes can be classified by the applied atmosphere used for pyrolysis (inert atmosphere) or combustion (oxidising atmosphere).

Pyrolysis

Pyrolysis is a thermal decomposition in which organics are cracked into a high carbon containing residue and a volatile phase. For cracking polymer chains a temperature of 500 °C to 800 °C is needed. The volatile phase is a mixture of gases and oils. Due to the reducing atmosphere metal components of the material are not slagged and can be recovered afterwards. The resulting carbonous residue is porous and brittle and can be easily cleaned of the metal surface. In a controlled process the glass and ceramic fraction of electronic scrap is unchanged. [8], [13]
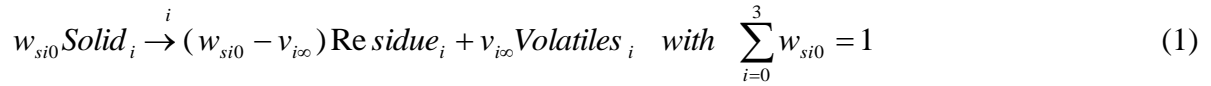
The investigations show that the atmosphere seems to have no influence on the cracking temperature, contrary to the content of bromine flame retardants. A higher heating range increases the gas and residue fraction. Gaseous products of the pyrolysis are methane, hydrogen bromine, carbon monoxide and dioxide. The form of bromine in the gaseous product is reported to be different in the investigations. Baronitini et al. only detected a small amount of hydrogen bromine in the off gas. In comparison to that Grause et al. detected a big amount of hydrogen bromine and Chien et al. found 72.3 % of all bromine as hydrogen bromine in the gas phase. [9], [10], [14]

In the residue both bromine free compounds like phenols and alkylphenols as well as bromine containing phenols and alkylphenols are found. The formation of polybromine dibenzodioxine and

dibenzofurane (PBDD/F) only takes place during combustion. [9] In a temperature range from 270 °C and 400 °C aromatic phenol products are built. [10]

To create a reliable kinetic model for the pyrolysis of electronic scrap the material has to be divided into three different fractions which are independent from each other and different in their behaviour. This separation has to be done due to the different organic compounds having individual cracking conditions. The kinetic model bases on the reaction which is shown in (1).

$$w_{si0} Solid_i \xrightarrow{i} (w_{si0} - v_{i\infty}) \mathrm{Re}\, sidue_i + v_{i\infty} Volatiles_i \quad with \quad \sum_{i=0}^{3} w_{si0} = 1 \tag{1}$$

i: number of fractions of the probe; w_{si0}: weight of the solid material; $v_{i\infty}$: maximum weight fraction of volatilised components in an infinitesimal time period; $Solid_i$: solid weight fraction; $Residue_i$: possible residue after the reaction, $Volatiles_i$: gases and volatilising materials from reaction i

The conversion coefficient α is a rate representing the weight of material which has reacted in a certain amount of time. It also can be described as a ratio of volatile components at the time t to the complete mass of volatile components which is generated from the starting material. The reaction speed is the temporal dependency of the conversion coefficient like it is shown in equation 2. [13]

$$\frac{d\alpha_i}{dt} = k_i \left(\frac{w_{si}}{w_{si0}}\right)^{n_i} = k_i (1 - \alpha_i)^{n_i} = k_i \left(1 - \frac{v_i}{v_{i\infty}}\right)^{n_i} \tag{2}$$

α: conversion coefficient; k_i: reaction rate constant, n: reaction order

By integration of (2) at known temperatures the conversion efficiency can be calculated. The relation between the conversion efficiency, the mass fraction of the residues and the volatile components is given by (4).

$$w = 1 - V = 1 - F_v \left(v_{1\infty}\alpha_1 + v_{2\infty}\alpha_2 + v_{3\infty}\alpha_3\right) \tag{4}$$

F_v= correction value

The correction value is needed because changing amount of inert material in each fraction. A variation coefficient VC is the difference between calculated and measured values. It is calculated using (5). The VC for the investigated trials by [13] of 2.5 % shows that equation 4 is a good approximation of a pyrolysis process. [13]

$$VC = \frac{\sqrt{\sum_{m=1}^{M}\sum_{j=1}^{N}\left(V_{m,j}^{\exp} - V_{m,j}^{cal}\right)^2}}{\overline{V}_{\exp} \cdot \sqrt{(N_{total} - P)}} \cdot 100 \tag{5}$$

VC: variation coefficient; N_{total}: number of data; P: number of parameters; v_{exp}: mean value of weight fraction of the volatile components; M: sum of trials; N: number of analysed parameters

To achieve a better approximation the accuracy of the reaction rate constant hast to be improved. J. Molto et al. introduced a correction of the reaction rate constant and Martín-Gullón et al. validated it which is shown in equation 6. [12], [13]

$$K_i^* = 0{,}64 \cdot k_i \qquad (6)$$

K_i^*: optimised reaction rate constant

Combustion

Like pyrolysis combustion is a thermal decomposition, but includes oxidisation. In a thermal gravimetrical investigation the thermal decomposition occurs as a heavy weight loss. The atmosphere has no influence on the thermal decomposition temperature. The oxidation reaction is accompanied with an additional weight loss. It is clear that the oxidation reaction is post combustion of the solid residue, because compared to pyrolysis there is no ash product. Only combustion leads to polybromine dibenzodioxines and dibenzofurans (PBDD/F). The creation of dioxins and furans takes place at the main decomposition process in oxidising atmosphere. The reason is the building of feedstock for dioxins or furans. Investigations with the flame retardant tetrabrominebisphenol A show, that the combustion up to 450 °C creates PBDD/F. [9]

The model with the three independent fractions valid for pyrolysis cannot be used for the combustion. To model the process procedure the individual fractions of the material have to be regarded. On the one hand these fractions react independently from each other. On the other hand these fractions can react independent from each other or sequency. Molto et al. investigated a reaction model of the combustion of electronic scrap containing organic compounds. He divided the material in five fractions which participate in three reaction steps, as described in the following: [13]

1. In the first reaction step only three pyrolysis reactions, described in (7), take place, where the oxygen partial pressure or concentration has no influence. The activation energy and reaction order for the three reactions are at the same level.

$$w_{si0} Solid_i \xrightarrow{i} (w_{solidi} - v_{i\infty}) \operatorname{Re} sidue_i + v_{i\infty} Volatiles_i \quad with \quad i = 1;2;3 \qquad (7)$$

w_{si0}: weight of the solid material; $v_{i\infty}$: maximum weight fraction of volatilised components in an infinitesimal time period; $Solid_i$: solid weight fraction; $Residue_i$: possible residue after the reaction, $Volatiles_i$: gases and volatilising materials from reaction i

2. Two of the in the first reaction step mentioned reactions create intermediate products which in the following subsequently react with oxygen like in (8). The activation energy and reaction order have the same level as for the pyrolysis. This reaction step adds four more reactions.

$$(w_{solidi} - v_{i\infty}) \operatorname{Re} sidue_{Ci} + O_2 \xrightarrow{i} (w_{solidi} - v_{i\infty}) \operatorname{Re} sidue_i + v_{i\infty} Volatiles_i \quad with \quad i = 1;2 \qquad (8)$$

3. In the third reaction step two of five fractions are oxidising by combustion without thermal decomposition. In that way the weight of the fraction is increasing. This reaction step adds two more reactions shown in (9).

$$w_{si0} Solid_i + v_{i\infty} O_2 \xrightarrow{i} (w_{solidi} - v_{i\infty}) \mathrm{Re}\, sidue_i \quad with \quad i = 4;5 \tag{9}$$
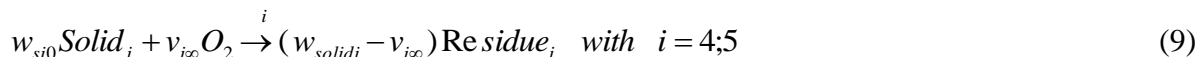
So all together nine reactions take place during combustion of electronic scrap. In that way it is clear that combustion is very complex compared to pyrolysis and to figure the development of a reaction model is a complex task. Molto et al. introduced an approximation of this process with a variety coefficient VC of 5.8 % which is shown in (10). [13]

$$w = 1 - V = 1 - F_P (v_{1\infty}\alpha_{1\infty} + v_{2\infty}\alpha_{2\infty} + v_{3\infty}\alpha_{3\infty} + v_{1cp\infty}\alpha_{1c\infty} + v_{2cp\infty}\alpha_{2c\infty} +$$
$$v_{1c\infty}\alpha_{1c\infty} + v_{2c\infty}\alpha_{2c\infty}) + F_M (v_{4\infty}\alpha_{4\infty} + v_{5\infty}\alpha_{5\infty}) \tag{10}$$

F_P: correction for the pyrolysed fraction; F_M: correction for the oxidizing metal fraction, $\alpha_{i\infty}$: conversion efficiency

Characterisation of electronic scrap

For the investigations two different kind of WEEE like printed circuit boards (later called L1) and a copper rich WEEE fraction (later called Y1) were used. Four different samples were taken in two different sampling processes for each kind of scrap. The average composition of L1 and Y1 is shown in table 1. The heating value of L1 is 9,400 kJ/kg and of Y1 11,100 kJ/kg. The composition of electronic scrap is very inhomogeneous.

Table 1:   composition of two types of scrap L1 and Y1

Scrap type	C	Cu	Si	Al	Fe	Ca	Sn	Pb	Ni	HA[1]
L1 in wt.-%	21,0	18,1	17,4	7,3	5,6	3,5	1,9	1,0	0,5	3,1
Y1in wt.-%	32,4	17,8	6,8	2,3	2,9	0,8	1,1	2,3	0,3	1,3

[1]: content of Halides as sum of Cl, Br, and F in the mixture

Calculation of WEEE combustion

The thermochemical calculations were done with the program FactSage™. For the combustion and the metal slag interaction calculations the databases Fact53, FToxid and FScopp were used. For the combustion a small amount of water is added to bring hydrogen into the system so that halide acids can be built.

The calculations of the combustion are also done for each type of scrap. For the scrap L1 the complete combustion takes place at 0,254 gO_2/g_{L1} and for Y1 at 0,54 gO_2/g_{Y1}. For L1 the metal yield is calculated as 23.6 wt.-% and for Y1 28.2 wt.-%. The compositions of the metal phase are directly

calcuelated from the FactSage^{TM} data. The metal phase calculation is in table 2 and the slag phases in table 3.

Table 2:   composition of the metal phase determined by combustion calculation

Scrap type	Cu	Fe	Sn	Pb	Ni	Sb	Zn
L1 in wt.-%	74,9	10,7	7,8	1,7	2,0	2,1	0,3
Y1in wt.-%	78,9	10,4	4,9	3,0	1,2	1,1	0,2

Table 3:   composition of the slag phase determined by combustion calculation

Scrap type	SiO$_2$	Al$_2$O$_3$	CaO	FeO	MgO	PbO	Cu$_2$O
L1 in wt.-%	68,7	19,4	9,5	1,3	1,0	0,035	0,0097
Y1in wt.-%	76,1	15,2	5,9	1,1	1,5	0,10	0,0107

Investigations of the influence of the pretreatment of Scrap

The investigation of the pretreatment consists of three trial series. The behaviour during pyrolysis, combustion and direct melting was observed.

For all experiments the experimental setup is the same (presented in Fig. 2) with variation only in the size of the crucibles for different experiments. The sample is placed in a porous reaction crucible that is placed in protection crucible to protect the furnace when the reaction crucible breaks. This system is heated up by a resistance heated furnace. Gases that are necessary for pyrolysis (argon), combustion (air) or direct melting (oxygen) are introduced to the sample through an injection lance.
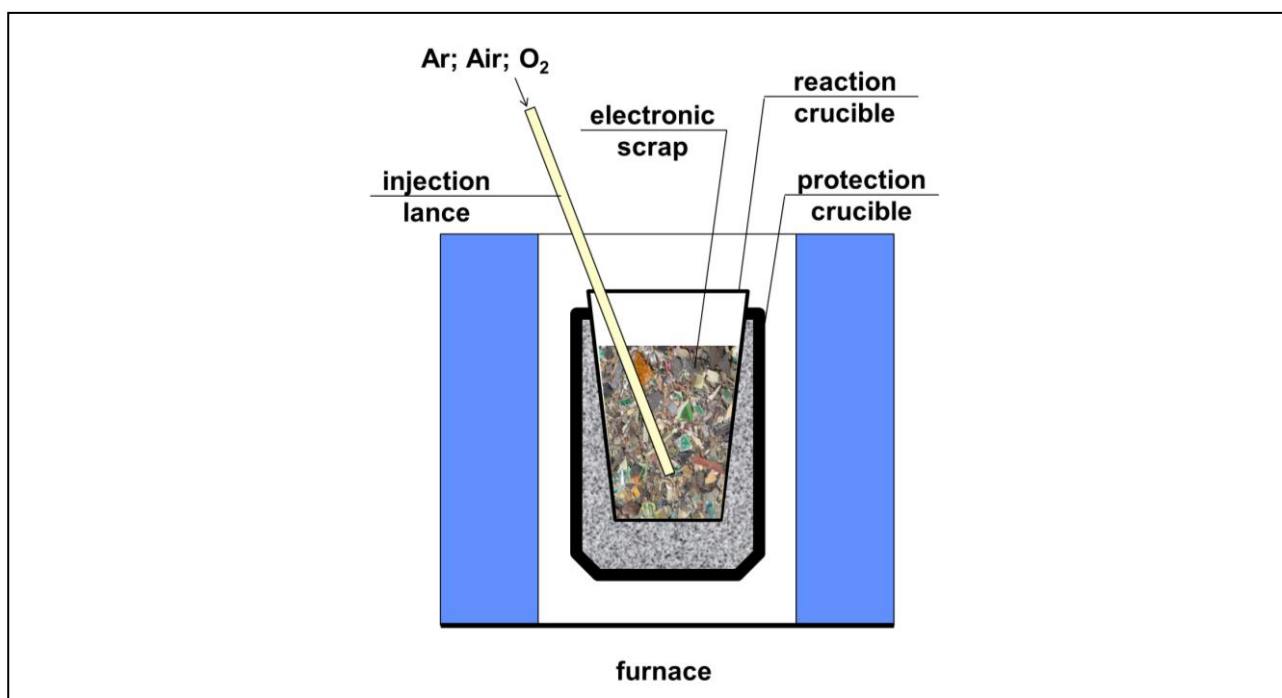
Figure 2:   Experimental setup

The pyrolysis trials are done in a resistant heated furnace in a 100-ml-scale. The crucible and the material are heated up to the process temperature. Pyrolysis works at a temperature of 600 °C. The furnace is flooded with protective gas (Argon) during the whole experiment. On the one hand the argon influent pipe is at the furnace bottom. On the other hand argon flows onto the material via a top lance. The duration of each experiment is set by the material. The experiment stops when the reaction is over by visible examination. Three experiments where done for each scrap type. After the pretreatment the material is melted in an induction furnace under argon atmosphere.

The combustion takes place at a temperature of 700°C which is above the incineration starting temperature of carbon. A resistant heated furnace in a 1-l-scale is used for these experiments. The combustion air is let into the material to be sure that all material is combusted and that no part only gets pyrolysed. Compressed air with a rate of 12 l/min was used. The experiments were stopped when the temperature of the mixture drops, because of the cooling effect of air without combustion. Three experiments where done for each scrap type. After the pretreatment the material is melted in an induction furnace under a constant air flow to burn left carbon.

The trials with direct melting were also done in a resistant heated furnace. The maximum temperature is at 1150 °C. The oxygen for this kind of trials is led through an alumina pipe and its throughput is controlled by Aalborg GFC37. The crucible is heated up to a temperature of about 800 °C. The oxygen is switched on and the material is charged to the crucible at once. In that way the pyrolysis of the material which takes place at lower heating rates is disabled. Due to the combustion of the organic material the temperature raises up to 1400 °C in hot spots.

In the direct melting process the off-gas (Fig.3) shows that at the beginning the combustion is incomplete because a lot of soot is built which is indicated by the dark colour of the off-gas. At the

end of the process the off-gas is white which indicates a complete combustion and converting of the metal phase.

Figure 3:   Off-gas from the direct melting process at the beginning (left) and at the end (right) of the experiment

In the direct melting process oxygen has two main tasks: the first one is the combustion of the carbon containing material after a thermal decomposition which produces energy for melting the material; the second one is a converting step of the metal phase. It is not possible to determine whether these processes run simultaneously or one after another.

To analyse the trials a mass balance and a chemical analysis of the metal and the slag phase was conducted. The off gas could not be analysed in mass or composition.

The average phase composition for the pretreatment modes, direct melting and calculation for each scrap type and an average are shown in Fig. 4.
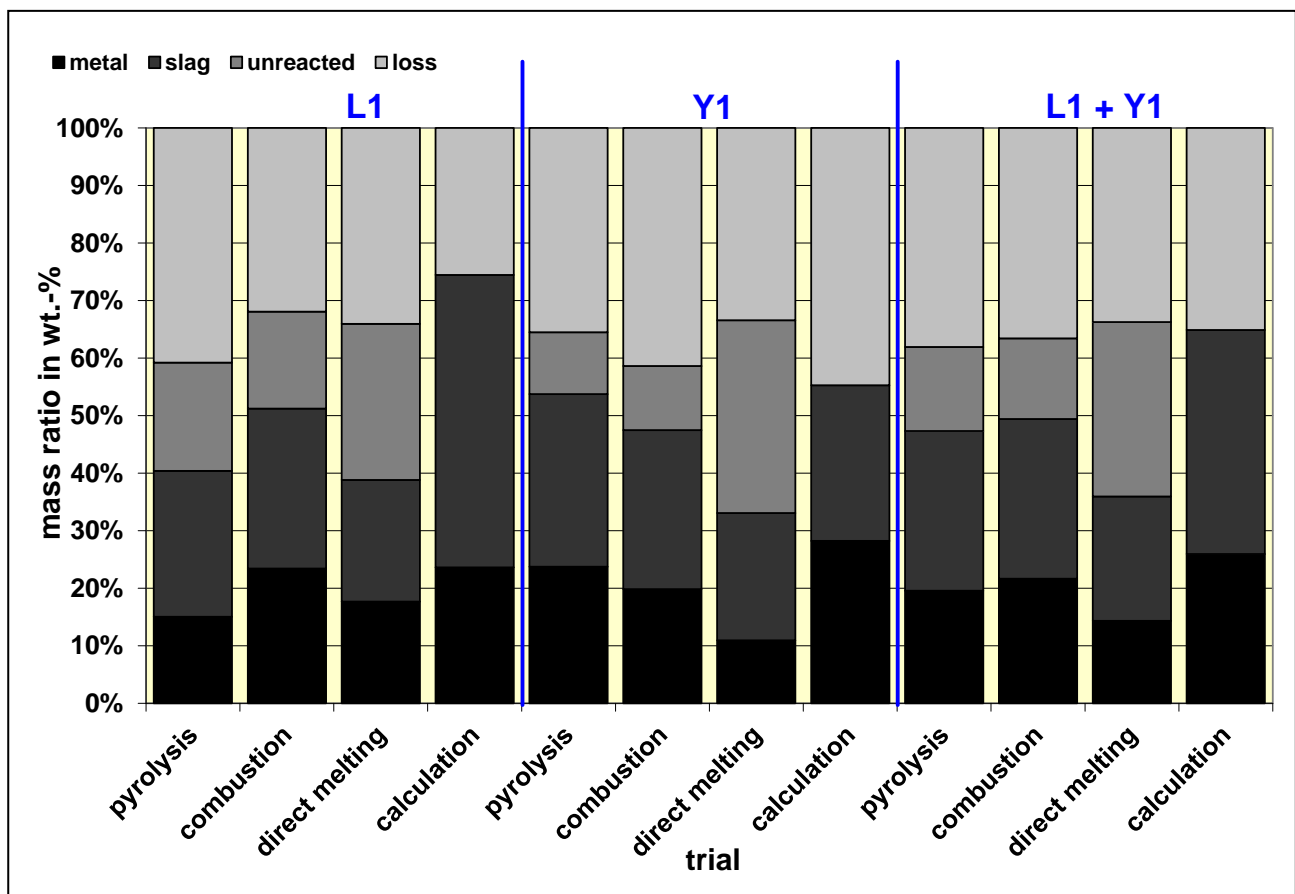
Figure 4: phase composition of the different pretreatment modes and the calculated values

In every experiment is more or less unreacted material on top of the slags. The highest amount of unreacted material is in the direct melting process. Also the pyrolysis process has a high amount of unreacted material. In this case the high carbon content in the material inhibits coagulation and settling down. Although manual feeding is done for the direct melting some material is not reacting with oxygen. This circumstance can be eliminated by a continuous stirring. A complete reaction of the material will have a positive effect on the metal yield. In most cases the ideal calculated metal yield is not reached. One reason is the unreacted material where some metal is left. Second reason is that for Y1 the slag phase has a higher amount. In that way more material is slagged. A third reason is the short settle down period. Some metal droplets cannot settle down and freeze in the slag. For L1 the loss via off gas is higher than calculated. In the first part of the process, the thermal decomposition, more volatile components are built.

The average composition of the metal and the slag phase which are formed in the direct melting trails are shown in table 4 and 5. For both scrap types the average composition is calculated. For the metal phase it is really difficult to get a uniform sample. The metal phase consists of large regions of high iron containing parts.

Table 4:   average composition of the metal phase from the direct melting trials for each scrap type

Scrap type	Cu	Fe	Sn	Pb	Ni	Sb	Zn
L1 in wt.-%	80,5	3,9	7,4	2,6	2,4	1,6	0,7
Y1in wt.-%	83,6	3,5	4,1	4,7	1,5	0,9	0,5

Compared to the calculated composition (table 2) the concentration of tin, lead, nickel, antimony and zinc is similar. There are differences in the concentration of copper and iron. Due to the higher concentration of copper and lower concentration of iron the content of oxygens in the raw material is higher than expected. Also a part of the injected oxygen is used for converting the metal phase. In that way a cleaner copper is produced. But to use the produced copper phase as feedstock for the normal anode furnace it still has to be treated furthermore.

Table 5:   average composition of the slag phases for both scrap types

Scrap type	SiO$_2$	Al$_2$O$_3$	CaO	FeO	MgO	PbO	Cu$_2$O
L1 in wt.-%	45,0	16,6	6,3	8,9	0,5	0,9	3,3
Y1in wt.-%	47,8	15,0	4,2	5,0	1,1	2,1	5,1

The slag compositions differ a lot from the calculated results. The content of silica is one third lower. Also alumina for L1 is 20% lower. The FeO content is higher than calculated but still lower than normal fayalitic slags. Calcia is within the optimal range of 3 to 8 %. Due to the converting subprocess during melting the copper content of the slag is very high. Also in a very reductive process that is reached by sub stoichiometric oxygen input, the copper content in the slag. The real compositions compared to the calculated slags have a lower viscosity. But the viscosity is still so high that some metal droplets cannot settle down. In that case lowering the viscosity increases the metal yield.

In Fig. 5 and Fig. 6 the influence of the specific oxygen mass and volume flow on the metal yield is shown for both scrap types.
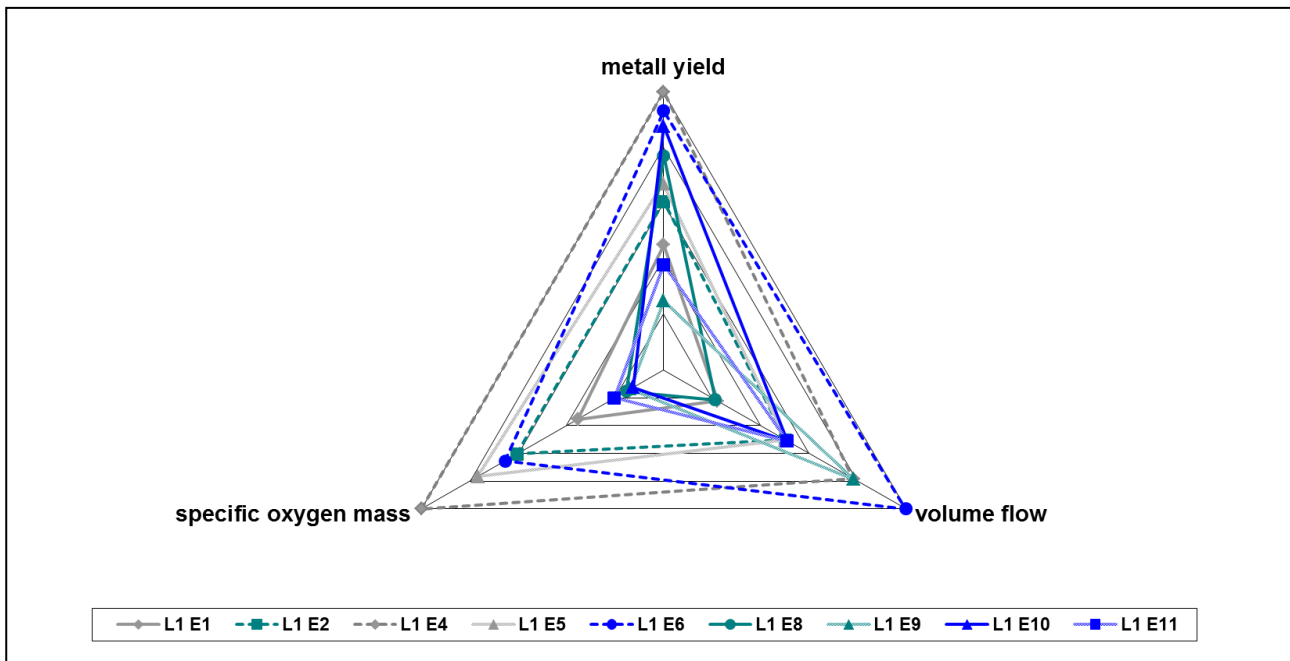
Figure 5:   Influence of oxygen input and volume flow on the metal yield for scrap type L1

The trials show that a high metal yield is possible to obtain with high and low oxygen input. The trials L1 E4, L1 E6 and L1 E10 achieve nearly the same metal yield with different oxygen input. With a higher mass of oxygen as input the volume flow has to increase. So with a higher volume flow the efficiency of oxygen decrease. That's why more oxygen has to be used in that case.
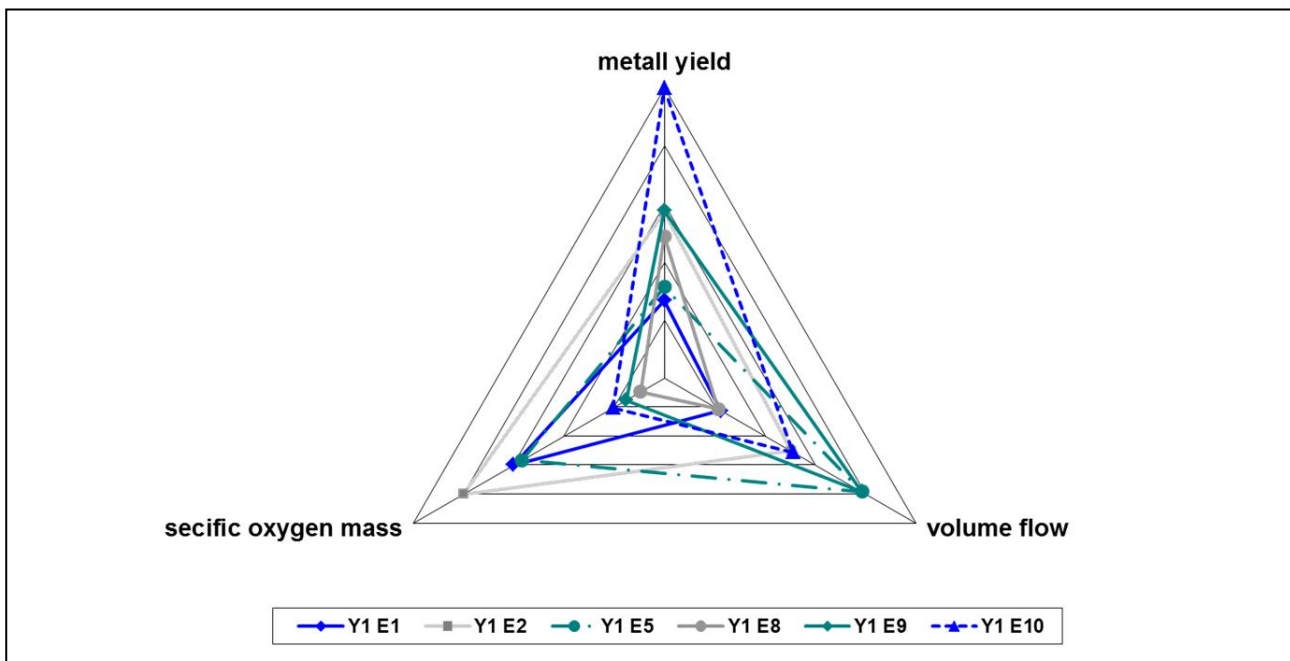

Figure 6:   Influence of oxygen input and volume flow on the metal yield for scrap type Y1

Trials Y1 E1 and Y1 E5 were done with the same oxygen mass input but very different volume flows. Both trails achieved the same metal yield. In the comparison of trials with the same volume flow is the higher metal yield in this trial which has the smaller oxygen mass input.

Comparing the metal yield two used scraps, fractions L1 achieved a higher metal yield than Y1. In the contrary to this, the thermodynamic calculations expected a higher metal yield for Y1. A reason for the lower yield for Y1 is the higher carbon content in the raw material. Some carbon can hardly be burned and increase the viscosity of the slag phase which avoids coagulation and settling down of metal droplets. The investigations show that by postcombustion and mixing in an inductive furnace the metal yield can be increased.

In a next step the influence of additions like magnesia, iron and other fluxes on the metal yield and slag composition will be investigated.

Trial setup 1-t-scale

At the IME a (T)BRC was built. The furnace is equipped with a 0.5 MW burner, an oxygen lance with a maximum flow of $100 \, Nm^3$, automating charging system which allows charging during operation. The burner can be changed from a gas/oxygen to a gas/air burner. The oxygen lance can work as a top lance or submerged. The rotation speed of the furnace can be changed between 0 and 10 rpm. The tilting angel of the furnace can be adjusted at any position. A picture of the furnace is shown in Fig. 7.
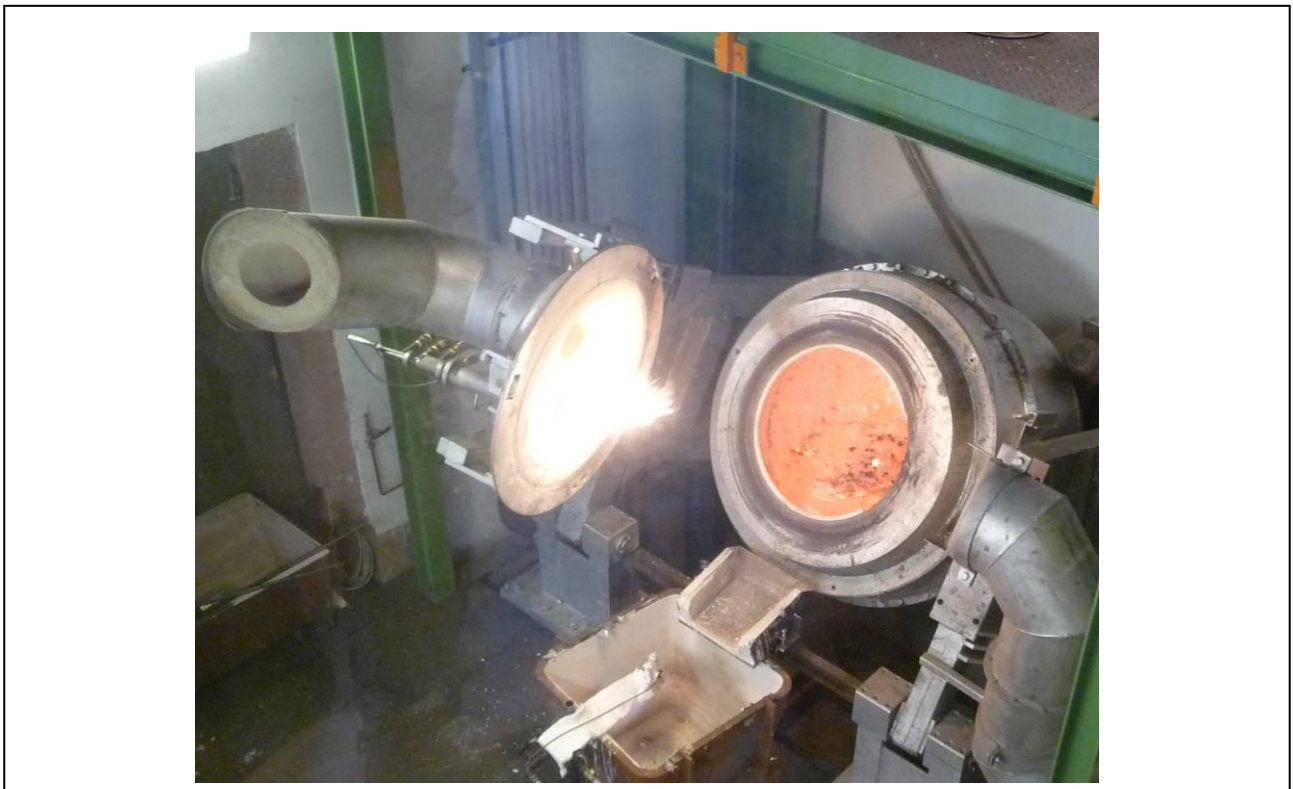

Figure 7: picture of the TBRC at IME

The Off-Gas is cleaned by a state of the art system which makes it possible to run this furnace in the heart of Aachen. Beside the process gas a secondary off-gas is sucked of a house in house construction around the TBRC. The primary process off-gas is cleaned by a hot ESP. Harmful volatilised elements are bonded to an adsorbent material with lime hydrate which is injected to the off gas system and separated in the ESP. The secondary off-gas is cleaned by a bag house filter. Both types of off-gas are together put into a washer to achieve very low pollution limits. The clean gas is in line controlled for most of the harmful elements.

The "warming up" of the furnace begins with a trial of pre-combusted material to learn about the handling and the behaviour of the furnace. After this trial PE and PP are added to investigate the autothermal characteristics of the process without the danger of the volatilization of harmful elements. In a last step the two types of electronic scrap, which were investigated in the small scale are planned to be recycled in this process.

The trials take place at a temperature of ca. 1250 °C. The material is fed to the furnace with a maximum rate of 120 kg/h.

Acknowledgment

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