Treatment of Industrial Lead and Zinc Slags in a Pilot Scale SAF

Jorn Bohlke, Bernd Friedrich, Erik Hecker

In the course of the pyrometallurgical production of lead and zinc it is unavoidable that significant amounts of those metals are contained in the slags which are produced as a by-product. These contents of lead and zinc in the slags lead to a decrease in yield and in the economy of the production and present a latent danger to the environment. Therefore, the treatment of these slags in a submerged arc furnace aiming at the recovery of zinc and lead and production of a useable slag is being investigated at the IME Process Metallurgy and Metal Recycling in a project which is financed by the German Federal Ministry of Education and Research. The investigations conducted include chemical analysis, thermodynamic modelling and experimental test work in a laboratory scale submerged arc furnace and a pilot scale submerged arc furnace. The aim of the project is to produce slags which can be utilised in further applications instead of having to be dumped, and at the same time increasing the efficiency of the lead and zinc operation by recovering lead and zinc from the slags.

Keywords:
Lead – Zinc – Slag – SAF, EAF – Hollow electrode

1 Introduction

Several pyrometallurgical processes are applied for the production of lead and zinc. These processes base upon different principles, for example shaft furnaces (Imperial Smelting furnace and lead shaft furnace) and bath smelting processes (e.g. QSL, Kivcet, ISA- and AUSmelt), they all have their individual advantages and disadvantages. An accepted disadvantage that is common for all pyrometallurgical processes is the fact that they produce a slag-product which inevitably contains some amount of the value metals. This entrapment, in this special case of lead and zinc, cannot be completely avoided because of thermodynamics, kinetics, limitations of the process technology and due to reasons of profitability of the production. Table 1 exemplary gives a list of published lead and zinc contents of industrial slags. The variations in the lead and zinc contents are caused by differences in process conditions, such as feed material, plant utilisation, technical equipment or operation mode. It is noteworthy that within the so called direct winning processes, as they combine oxidation and reduction in one reactor, a lead and zinc rich slag is obtained either locally inside the reactor (e.g. QSL or Kivcet process) or temporarily as intermediate product which is treated further in a subsequent step in the same or another reactor (e.g. ISA- or AUSmelt process).
The entrainment of lead and zinc in the slags may be tolerated because of the above-mentioned reasons, but nevertheless it leads to undesired consequences. The immediate consequence for the producer is that lead and zinc in the slags mean a loss decreasing the metal yield of the operation. Another consequence is that a too high lead and/or zinc content in the slag can result in a violation of limit values given by environmental and legislative regulations, so that the slag cannot be used as construction material in roadwork construction or the like. Last but not least, heavy metals in slags may represent a latent risk for the environment, for example if they are soluble in ground water. In the worst case the slag has to be dumped, which in term causes further cost and decreases the economics of the operation even more. Therefore a treatment of the slags is reasonable and recommendable. A suitable slag treatment can increase the metal yield and the profitability of the production and safeguard a slag available of decreased volume and improved quality, which allows further use in other applications.

At present there are some processes employed for this purpose such as slag fuming [4], the ISA- and AUSmelt process [5], the Plasmazinc process [6] or the Enviroplas process [7]. Most of these processes are designed for rather constant conditions and display disadvantages in flexibility and efficiency. A very flexible reactor for a slag treatment is the submerged arc furnace (SAF) which permits high temperatures, low off gas volumes and an efficient use of energy. A SAF is characterised by a compact and flexible design so that it can be integrated almost into every existing operation. Furthermore the SAF technique provides the possibility to treat liquid material in-line as well as solid materials when the circumstances allow or demand it. On the other side the SAF technology has the taint of high investment costs and a high demand for usually rather expensive electrical energy.

SAF technology is proved already in slag treatment for copper smelting, and in the course of the research project the treatment of slags from different lead and zinc production sites in a SAF is investigated. The proposed process bases on the carbothermic reduction of lead and zinc oxides in the liquid slag at high temperatures. Zinc ought to be fully vaporated from the slag and can be recovered in the flue dust, whereas lead is expected to be found partly in the flue dust and partly as liquid metallic lead in the furnace. As side effect the reduction of iron oxides supports the process by achieving lower slag viscosities and — according to Stokes’ Law — higher settling velocities [8]. As target values for the final contents in the slag 0.1 % fox lead and 1 % for zinc have been set. The focus of the work therefore is the investigation of the thermodynamical limits and the definition of the process window for maximum efficiency by using a submerged arc furnace for the slag treatment.

2 Investigated slags

In the course of the research work slags of different lead and zinc production processes have been treated at IME Process Metallurgy and Metal Recycling, RWTH Aachen University’s department for non-ferrous metallurgy. The investigation focuses on two types of slags which differ mainly in the lead, zinc and sulphur contents ("slag 1" and "slag 2"). Figure 1 shows the average compositions of these slags as they have been calculated upon the basis of chemical analyses.

The average lead content is 1.5 % in slag 1 and 4.4 % in slag 2, according to literature and own investigations about 80 % of the lead is present in metallic form [9]. The average zinc content amounts to 11.1 % in slag 1 and to 12.8 % in slag 2. Slag 1 contains more sulphur (in average 2.6 %) than slag 2 (in average 0.2 %). This leads to a different zinc mineralogy in the slag as sulphur primarily forms zinc sulphide so that zinc sulphide and zinc oxide make up for approximately one half each of the total zinc content of slag 1, whereas in slag 2 zinc is present almost completely as zinc oxide. Besides the zinc and lead components a major part of the slags consists of the standard slag formers SiO₂, CaO, Al₂O₃ and MgO (slag 1: 47.6 %, slag 2: 43.1 %). Furthermore iron is present in the slags, the fraction of iron oxides is 35.7 % in both slag 1 and slag 2. Iron is an important component of the slags because of its nature as a transition element which can be exist as Fe₀, Fe²⁺ or Fe³⁺, so that it can be reduced or act as reducing agent itself. In addition to this, the ratio of Fe³⁺/totFe⁺ takes influence on the viscosity of the slag (see above). As iron can be potentially reduced to the metallic state if the zinc content gets low, it is essentially for the slag treatment process that this iron phase can be easily molten during or after slag treatment, which is an advantage of the SAF technology.

In addition to slag 1 and slag 2 test runs have been carried out with an intermediate product of a lead winning process ("slag 3") in order to investigate the treatment of lead-rich slags in a submerged arc furnace. This allows the tests in a range of slag compositions as wide as possible. Slag 3 contains 55.3 % lead and 6 % zinc, the standard slag

### Tab. 1: Published lead and zinc contents of Pb-Zn slags [1-3]

<table>
<thead>
<tr>
<th>Process</th>
<th>Average lead content (%)</th>
<th>Average zinc content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb shaft furnace</td>
<td>1.5 to 2.5</td>
<td>&lt; 18</td>
</tr>
<tr>
<td>IS furnace</td>
<td>1 to 3</td>
<td>6 to 7</td>
</tr>
<tr>
<td>QSL process</td>
<td>2 to 5</td>
<td>7 to 15</td>
</tr>
<tr>
<td>Kivet process</td>
<td>4 to 5</td>
<td>9 to 18</td>
</tr>
<tr>
<td>Issmelt process</td>
<td>&lt; 3</td>
<td>8 to 12</td>
</tr>
</tbody>
</table>

Fig. 1: Average composition of investigated slags: slag 1 and slag 2
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All slags that have been treated in the SAF had been provided in water granulated form, so that the particle size was about 0.5 to 5 mm. Figure 2 gives an impression of one treated slag.

3 Thermodynamical modelling

In advance of the experimental test work thermodynamical calculations have been carried out for slag 1 and slag 2. The aim of these calculations has been to obtain information about the effect of process parameters on the lead and zinc recovery and about the theoretical maximum recovery of lead and zinc from those slags. Since thermodynamical calculations only consider equilibrium states and neglect kinetics, the obtained values can be seen as limits for the slag treatment and thereby help to assess the results of the experimental test work. The thermodynamical modelling was done with the FactSage® software-package [10]. FactSage® has proven to be useful for this purpose because it offers databases which include liquid metal and slag phases as well as solid oxides and sulphides. For the liquid slag phase, subsystems containing PbO and ZnO have been optimised by the FactSage® developers, so that the results of the calculations can be expected to be reliable. The main parameters that have been examined in the course of the thermodynamical modelling are the following:

- input material: slag 1 or slag 2
- reducing agent: effect of carbon addition (relative to the amount of lead and zinc)
- effect of atmosphere (0, partial pressure): air or argon
- temperature: from 700 °C up to 1500 °C

Figure 3 and Figure 4 exemplarily show calculated equilibria for slag 1 with addition of carbon under air atmosphere. From Figure 3 it can be seen that solid oxidic phases are stable up to approximately 1130 °C and that zinc sulphide (sphalerite and wurtzite) is present even up to approx. 1330 °C. The smooth transition from sphalerite to wurtzite can be seen as indication for a correct calculation. A first liquid slag phase is formed at 1050 °C and its proportion rises strongly in mass up to 1150 °C where it makes up approximately 90% of the solid and liquid phases. Figure 4 shows the corresponding gas phase for the same calculation. Because of the addition of coke carbon monoxide and carbon dioxide are dominant in the gas phase at lower temperatures, with carbon monoxide getting more stable with rising temperatures. At temperatures above 900 °C zinc oxide is reduced in the slag and metallic zinc is evaporated as expected into the gas phase, so that a part of the carbon monoxide is turned into carbon dioxide. At higher temperatures zinc oxide and zinc sulphide react jointly and form gaseous zinc and sulphur dioxide. This effect is much more prominent than the carbothermic reduction of zinc, so that in slag 1 sulphur will act as the important reducing agent for zinc. It can be shown simultaneously that for slag 2 (where sulphur is not significantly present) carbon and iron together reduce zinc at temperatures above 800 °C.
Figure 3 and Figure 4 can serve as examples for the thermodynamical modelling results. Figure 5 and Figure 6 summarise the outcome of this investigation regarding the effect of the carbon addition and of the atmosphere on the lead and zinc recovery from the slags. It can be seen that the addition of carbon and the use of an inert (argon) atmosphere that restricts the access of oxygen to the slag improve the evaporation of lead and zinc from the slags into the gas phase. From the thermodynamical point of view it is possible to collect close to 100% of the lead and zinc content of the slags in the gas phase (technically seen, as flue dust). In theory suitable conditions for the slag treatment can be generated by the use of carbon, a high temperature (more than 1500 °C) and the exclusion of oxygen in the atmosphere. For this set of parameters slag 1 and slag 2 contain theoretically only 0.01% respectively 0.03% lead and 0.21% respectively 0.24% zinc.

As mentioned before, the thermodynamical calculations by definition neglect kinetic effects. A short consideration of kinetics is given elsewhere [11, 12]. Due to this negligence of kinetics it is not possible to get information about the time necessary to establish the equilibrium state. Furthermore some parameters used for the calculations are idealistic or based upon assumptions (for example the ratio of the amount of feed slag to the amount of atmosphere or the composition of the atmosphere) and other parameters cannot be maintained in practice with perfect accuracy (e.g. temperature). Therefore the achievable results in practical test work can differ significantly from the results of the modelling. But particularly the imponderability of real processes makes the thermodynamical modelling more useful in the way that the results of the experimental test work can be evaluated by comparing the obtained results with the theoretical optimal achievable values.

4 Experimental

In order to investigate the carbo-reduction treatment of lead and zinc bearing slags in a submerged arc furnace, several tests have been carried out. A series of test runs in a 100 kW laboratory scale SAF of the IME served to give first results as preparation of the pilot scale test work [12]. The focus of the entire test work was on the treatment of the slags 1 and 2 and as well of the lead-rich intermediate product in the pilot scale SAF of the IME in order to define...
a set of process parameter serving quick operation and lowest Pb/Zn-contents in the slag.

4.1 Equipment: Pilot-scale SAF at IME

A 500 kW pilot scale SAF, electric power provided up to 5.3 kA and 110 V in DC mode, was used for the experimental validation of the theoretical results. The shell of the furnace is a water-cooled double steel wall and is lined with refractory bricks (chrome-magnesite) on the inside establishing an inner diameter at the bottom of 520 mm. The inner height of the furnace is 900 mm, which leads to a total volume of 250 litres. The furnace is equipped with a water-cooled copper bottom electrode and a graphite top electrode which is 150 mm in diameter. Graphite electrodes can be full or hollow electrodes, the latter having a 65 mm hole for the charging of fine materials into the electric arc zone. It is possible to tap melt from two tap holes, one on bottom level and one 500 mm above bottom level. The whole furnace is housed in order to collect the entire off gas which is gathered at 5000 m³/h and cleaned in a bag filter, where flue dust is collected for weighing and sampling, and subsequently in a gas scrubbing unit. A PC-based data logging device automatically saves important parameters such as several temperatures of the furnace, cooling water and off gas system and electrical data for later analysis. Other parameters like the temperature of the melt have to be measured manually and discontinuously.

The pilot scale SAF has been used also for different purposes, like recycling of different types of batteries (NiMH, ZnC, Alkaline), production of ferro-chromium and ferro-silicon, treatment of PGM containing slags, ilmenite reduction or the investigation of the fusibility of different kinds of ores. Temperatures of more than 1800 °C are possible. Figure 7 shows an overview of the pilot scale SAF at IME.

4.2 Procedure

The treatment of industrial lead-zinc slags in the pilot-scale SAF has been carried out batch wise in DC mode with the copper bottom electrode acting as anode. In each test run approximately 250 to 300 kg of water granulated slag have been molten and treated, the tap-to-tap time was 2 to 2.5 hours. The test runs have been conducted in series of three or four in order to gain comparable and reproducible results. Before each first test run of the day the furnace was heated by smelting 150 kg of steel scrap for 2 hours, with the intention of preheating the refractory lining. In total more than 5.6 tons of lead-zinc slag have been molten. The charging of the feed material takes roughly 1 hour of the total time and is done with a power input of up to 350 kW. After or already during the charging of slag lignite coke (particle size: 1 to 5 mm) is added to the melt using a small amount of nitrogen as carrier gas through the hollow electrode. The amount of added coke was calculated on the basis of the lead and zinc contents of the slags with 20 % stoichiometric coke-excess. A temperature of 1500 °C or more was aimed at, especially in preparation of tapping. The temperatures of the melt have been measured discontinuously by a lance. Several samples of the liquid slag are taken during a test run, either by inserting a cold iron bar into the melt or by using a scoop for extracting a larger portion of the melt. After a holding
time of about half an hour the treated slag is tapped into ladles and the next test run can begin. During the tapping of the slag a small portion of the slag is granulated in a water bath. The produced slag, the collected flue dust and where possible the produced metal are weighed with the intention to put up mass balances. Samples are taken from the water granulated final slag, the flue dust and if possible the metal and analysed by ICP and X-ray fluorescence analysis.

4.3 Results

The lead and zinc contents of the slags both decreased during the treatment in the SAF, but the decrease of lead and zinc show rather different character. Figure 8 shows the development of the lead content during a test run for slag 1 and slag 2 exemplarily. As can be seen for the given test run lead is removed noticeably from the slag right from the beginning. This can be seen as a proof for the presence of metallic lead in the slag which does not need to be reduced but only has to melt and to settle out of the slag phase into a lead phase. The trend of the lead content is similar for slag 1 and slag 2, but in the former the effect is more significant because of the higher initial lead content. The results of the treatment of the lead rich intermediate product (slag 3) confirm this behaviour. The treated lead rich slag had an initial lead content of 55.3% which leads to a large metallic lead phase settling at the bottom of the furnace. It has been observed that this can lead to an extensive infiltration of the installed refractory lining, in industrial practice this can be avoided by using more appropriate refractory materials, for example carbon bricks. A separate iron layer was not found, reduced iron-droplets were only found in the final slag as disperse droplets.

The behaviour of zinc is not identical to lead. From Figure 9 it is seen that the removal of zinc from the slags does start only delayed at the beginning of the SAF-treatment but accelerates at its end. This can be ascribed to the fact that zinc is completely bonded as oxide or sulfide in the slags so that it has to be reduced and evaporated for removal. This requires higher temperatures in the melt than the melting of lead, temperatures which are difficult to achieve during the charging period. Whereas the reduction of zinc is inhibited during the charging of the feed slag, it increases and leads to quickly decreasing zinc contents when homogenous conditions and higher temperatures in the melt are achieved.

One decisive factor for the success of the treatment of lead-zinc slag in an SAF is the final content of lead and zinc in the slags after the treatment, because this determines whether a slag complies with regulations. It also is related to the efficiency and yield of the process. In the conducted test work the majority of the test runs produced very good results which are in agreement with the targets (set limits) of max. 0.1% lead and 1% zinc. This is shown in Table 2. The achieved minimum lead contents of the slags were analysed as 0.02% for slag 1, 0.08% for slag 2 and 0.29% for slag 3. For slag 3 a high value of the maximum lead content of the treated slag was determined, but this is falsified by the metallic lead phase in the furnace.

The minimum zinc contents of the final slags were analysed as 0.16% for slag 1, 0.72% for slag 2 and 0.07% for slag 3. Comparing the experimental values to the calculated data, it can be seen that the minima meet the prediction of the thermodynamical calculation very good. In the case of slag 2 the zinc content went even below the prediction which can be explained by a higher mean temperature in the test run than assumed in the calculation. A positive side effect of the carbo-reduction treatment of the slags is the diminishing of the slag amount. For slag 1 and slag 2 the quantity of slag can be reduced up to 16%, in the case of slag 3 this decrease even amounts to 54% to 70%. The reduction of the mass of the slags is important to minimize the cost for further use or landfill.

Furthermore Table 2 shows the lead and zinc contents of the gained flue dusts. For slag 1 and slag 2 the zinc content exceeds the lead content whereas for slag 3 due to the high Pb/Zn-ratio in the input material the dust product contains much more lead than zinc. The ratio of produced flue dust to the amount of treated slag varies between 6.5% and 13.5% for slag 1 and 2 and between 12.5% and 13.5% for slag 3. The produced flue dusts may be easily processed for lead and zinc winning in other processes.

Figure 10 and Figure 11 exemplarily illustrate the distribution of lead and zinc among the products of a test run for
slag 2 and slag 3. As slag 3 originally is rich in lead a metallic lead phase is formed whereas the treatment of slag 1 and slag 2 rarely produces a metallic phase due to the lower lead contents.

In the case of slag 2 (Figure 10) 94.7 % of the lead is transferred from the initial slag to the flue dust, only 1.1 % of the lead remains in the slag phase. As no metallic phase could be separated after the test run, 4.2 % of the lead has to be considered as a loss which is assumed to be caused by diffuse emission and, more important, smallest droplets of metallic lead that did not settle or infiltrated into the refractory lining of the furnace. Zinc on the other side is transferred to 76.6 % into the flue dust, 5.6 % remain in the slag phase. The loss of zinc adds up to 17.8 % and is higher than the loss of lead. Here the most probable cause for the loss of zinc are diffuse emissions of zinc oxide in the off gas. As zinc is reoxidised from metallic, gaseous zinc to zinc oxide in the off gas by secondary air, the particle size of the zinc oxide in the flue dust is assumed to be very small, so that it is not collected entirely by the bag filter but partly by the gas scrubbing system.

Figure 11 shows that 59.2 % of the lead content of the initial slag 3 is collected in the metal phase and 16.9 % of the lead content remains in the treated slag phase. From optical inspection it can be assumed that lead in final slag phase is still partly present as metallic droplets that did not settle into the metal. Another 14.4 % of the lead content of slag 3 can be found in the flue dust, and 9.5 % must be considered as loss in this test run. A main part of this loss is assumed to be caused by infiltration of the refractory lining. For zinc the distribution is quite different. Here 70.7 % of the zinc content of the feed slag is collected in the flue dust and only 0.4 % of the initial zinc content remains in the slag phase. As zinc is not included in the metallic phase, this leads to a mass balance loss of 28.9 %. The loss of zinc is assumed to have been caused as stated above for slag 2.

Other important factors for the economy of a slag treatment process are the energy consumption and the coke consumption. The energy input into the furnace has been approximately between 950 kWh/t slag and 1350 kWh/t slag for slag 1 and slag 2, and between 1100 kWh/t slag and 1250 kWh/t slag for slag 3 respectively. The variations are due to differences in the average temperature and duration of a test run. It has to be noted that the amount of energy losses that are caused by cooling water and other effects cannot be quantified and therefore are included in these values as well as the energy that is needed to melt the solid feed material. Consequently the necessary energy for the energy optimised inline treatment of liquid slags in a SAF will be considerably lower than these given values. The energy loss by cooling water will be measured during future test runs in order to make more exact data accessible. The average temperatures during the test runs have been in the area of 1350 °C to 1550 °C. The applied coke consumption was about 30 kg/t slag for slag 1 and slag 2 and about 50 kg/t slag for

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**Tab. 2: Lead and zinc contents in the slag product after SAF treatment compared to modelling (minimum contents) and in the flue dust after SAF treatment (maximum contents)**

<table>
<thead>
<tr>
<th>Slag type</th>
<th>Slag product (minimum)</th>
<th>Modelling*</th>
<th>Flue dust (maximum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pb 0.02 %</td>
<td>0.01 %</td>
<td>37.2 %</td>
</tr>
<tr>
<td></td>
<td>Zn 0.16 %</td>
<td>0.21 %</td>
<td>77.8 %</td>
</tr>
<tr>
<td>2</td>
<td>Pb 0.08 %</td>
<td>0.03 %</td>
<td>38.1 %</td>
</tr>
<tr>
<td></td>
<td>Zn 0.72 %</td>
<td>0.24 %</td>
<td>70.5 %</td>
</tr>
<tr>
<td>3 (lead rich)</td>
<td>Pb 0.29 %</td>
<td>not calculated</td>
<td>56.3 %</td>
</tr>
<tr>
<td></td>
<td>Zn 0.07 %</td>
<td>not calculated</td>
<td>27.6 %</td>
</tr>
</tbody>
</table>

* calculated at 1500 °C, argon, carbon addition
slag 3, but in all the conducted test runs a coke excess has been used which can be minimised in practical operation.

**Summary and outlook**

Due to reasons of thermodynamics, kinetics and profitability of production, slags which derive from the pyrometallurgical production of lead and zinc always contain significant amounts of lead and zinc. Because of economical, ecological and legislative reasons a special treatment of these slags is recommended if a direct use e.g. in road construction is not possible and dumping of the slags is to be avoided. A technology which offers appropriate characteristics for a treatment of lead-zinc slags is the carbo-reduction process in a submerged arc furnace.

In a BMBF-financed project the treatment of lead-zinc slags in a submerged arc furnace has been investigated. Thermo-dynamical calculations have shown that sulphur and iron which are present in the slags act as reducing agents for zinc. Under suitable conditions both lead and zinc can be recovered theoretically almost to 100 %. Such suitable conditions are served by high temperatures, the addition of carbon as reducing agent and a non-reactive atmosphere. Subsequent experimental test work has been carried out in laboratory scale and pilot scale submerged arc furnaces. This test work proved that the SAF technique is very adoptable for a slag treatment by producing slags with very low lead and zinc contents and a flue dust rich in lead and zinc. The final lead and zinc contents in the slags in the majority of the test runs were in agreement with the target limits of 0.1 % lead and 1 % zinc in the slags. Thus the results of the thermodynamical modelling could be confirmed and it can be stated that kinetics do not completely prohibit the equilibration of the treatment. It has been found that the removal of lead from the slags differs from the removal of zinc. Whereas lead is removed rather quickly from the beginning of the process, the removal of zinc at first is inhibited and only increases when homogenous conditions and high temperatures are reached. Lead and zinc can both be recovered to between 70 % and 95 % in the produced flue dust and, if formed, in a metallic lead phase. This metallic lead phase does form significantly when the initial slag contains a large amount of lead. The produced flue dust can be used for recovery of zinc and lead by pyrometallurgical or hydrometallurgical means, the produced crude lead can be introduced in the lead refining operation. The SAF treatment can be applied to slags of lead and zinc winning processes as well as to intermediate products of lead winning processes which are rich in lead.

Further investigation will focus on the acceleration and intensification of the slag treatment in the SAF by pneumatic injection of coke into the melt instead of or additional to its feeding through a hollow electrode. The test work in this area is expected to be carried out in the course of 2005. At the same time the furnace will be equipped with additional sensors with the intention to observe the energy balance of the process. Furthermore the economical limits of the use of the submerged arc furnace will be calculated on the basis of the gathered data and by evaluation of the produced slag, flue dust and crude lead. On the basis of this a recommendation for the treatment of lead and zinc bearing slags will be given.

**Acknowledgment**

We thank BMBF (Federal Ministry of Education and Research) for financial contribution to the project.

**Literature**


