



Potential of Minimizing Magnesium Losses in Black Dross through Optimization of Salt Fluxes

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Abstract

Magnesium recycling is typically based on the use of fluxes, mostly containing MgCl_2 , KCl , NaCl and CaF_2 . A very common mixture in industry (“Flux 5”) has the role of separation of surface oxides, gases or other contaminations from metal. It is state of the art that application of salt flux in contact with magnesium scrap leads inevitably to the formation of a sludge so called “black dross” containing both metallic and oxidic magnesium as well as salt flux components. In this research project, it has been tried to investigate the influence of chemical composition of salt fluxes applied in magnesium recycling on the sludge formation degree with the aim of minimization of metallic magnesium loss in black dross. The results represent the general increasing effect of fluorine containing components as well as magnesium chloride inside fluxes. The value of 5 Wt % of fluoride components appeared to have the best effectivity on coalescence and CaF_2 is the best economical/technical choice because of its reasonable price against LiF and MgF_2 as well as its high coagulation effectiveness in comparison with KF and NaF .

Keywords: Flux, Salt, Coalescence, Black Dross, Magnesium

1 Motivation

In the magnesium recycling process, an unwanted sludge as a heterogeneous system composed of oxidic and metallic magnesium as well as salt combinations and impurities is formed inevitably due to the interfacial tension effects between metal, salt and oxides. Generally speaking, such sludge is formed when salt fluxes, which are used to release magnesium from scraps and to separate the surface oxides, other non-metallics, gases or further contaminations, combined with metallic particles as well as oxides. On the other hand, the capability of salt flux to coagulate metallic magnesium droplets is - beside interfacial tension - the most influencing parameter in formation of such unde-



sired dross. There is still no way to recover this dross economically or to reduce its formation. One part of an ongoing PhD-project, the optimization of salt flux chemical composition to achieve the most magnesium coalescence efficiency, is task at IME Institute of RWTH Aachen University. The second part, the development of a dross recycling concept, has been already published at the conference proceedings of IMMC 2008 Istanbul.

2 Background

The growth of magnesium use, the lightest structural metal, has been tremendous in the last years due to the increasing need for low-density structural materials for both air and land travel [1]. Beside the low density of about 1.8 g/cm^3 , further properties like good heat conductivity, high specific strength and excellent damping behaviour are the reason of the increasing application of magnesium as a basic material for light weight constructions [2]. Parallel to the rise of magnesium production as well as the usage of magnesium products the quantity of magnesium scrap increases too. Throughout the remelting process of magnesium scraps, the formation of dross or sludge is not evitable [2]. Melting of Mg-scraps is usually carried out on a batch or semi-continuous basis using a flux or suitable protective atmosphere to ensure that the molten magnesium does not react with air. Protecting molten magnesium has always been a key issue [3]. On the other hand, how to manage the scrap and by-products of magnesium will be a challenging question in future. The magnesium metal in all scrap types is valuable and techniques for its recycling are strongly under development [4]. Black dross, in some publications named as magnesium scrap “type 7” [5] is a by-product from magnesium smelting and recycling, which contains magnesium both in metallic and oxidic form as well as salt components [5-7]. Dumping or disposal of the dross or sludge in the ocean, which occur very often currently, is not a final solution in the matters of saving the resources and environmental aspects [2]. The metal value and the high cost for landfilling drives the developments to find suitable methods to recycle Black Dross as well as to prevent its formation [8].

Salt fluxes play a great role in the remelting of secondary aluminium and magnesium. A flux should cover the metal to avoid oxidation. It should also dissolve or suspend dirt, oxide and other dummy materials sticking to the metal [9]. Most of the recent research activities showed that the coagulation of aluminium droplets in a molten salt bath could have a direct relation with delamination of oxide layer [10,11]. Therefore, the third and better to say the most important requirement for a flux should be the promotion of coalescence of the molten metal to strip the adhering layer of oxide on the metal surface. Some physical requirements of a salt such as low melting point, low density, no reaction with or contamination the metal, low viscosity, easy separation from the metal, not attack the refractory walls and etc. can be determined through simple laboratory determination or evaluation of tabulated data. However, coalescence is the least understood and researched and is maybe the most difficult one to quantify [9].



The main goal of the work reported in this paper was to modify the chemical composition of salt flux used in magnesium recycling in order to minimize the possibility of dross formation during remelting process. The focus of this paper is on the coalescence behaviour of such salt compositions on magnesium molten droplets. Quite contrary to aluminium salt fluxes, whose coagulation's characteristics have been well investigated [12,13,14,15], the publications about magnesium salt flux development appear to be very poor and the modification of fluxes' chemical as well as physical properties have not been yet researched meaningfully.

Generally speaking, there is a variety of methods to measure the coalescence of salt fluxes in aluminium melting process. In all of them it has been proved that without a fluoride addition, it takes many hours to complete coalescence, but an addition of several weight percentages of fluoride containing salts is able to accelerate the coalescence by a factor of dozens or hundreds [12,13]. Furthermore, the addition of fluorides to the equimolar chloride mixture can decrease the interfacial tension between molten salt and aluminium too, which leads to more coagulation. Van linden [16] showed that aluminium recovery is more effective when the fluoride concentration is 10 mol%; however, Tenorio and Delgado suggested that only 2.5 mol% fluoride addition could be sufficient [13].

Lakaschenko and Slatkova tested the kinetics of coalescence of aluminium powder in a porcelain crucible in a retort. Salt flux and Al-powder were remelted firstly. After solidification the salt was leached and the Al-particles were divided into different grain sizes by sieving classification [17]. Peterson loaded 5g aluminium powder (1-2 mm diameter) in a molten salt bath and then observed the devolution of coagulation in a furnace equipped with transparent walls [9]. Ye and Sahai suggested the method of immersion two droplets of aluminium into molten salt without introduction of the size of droplets [18,19]. Sydykov applied a quartz crucible placed on the bottom of an electrical furnace with a slope of 20° to the horizontal plane. He fed 200 Al-spheres of same diameter into the crucible with 5g molten salt flux. After a given coalescence time and subsequently solidification, the received material was leached with water and Al-globules were sorted manually. Globules of each size fraction were counted and weighed [17].

3 Methodology for Selection of Magnesium Salt Fluxes

The salt fluxes applied in this work, were chosen based on the chemical composition of the common used fluxes in magnesium recycling processes. They included MgCl₂, KCl, NaCl, and CaF₂. A matrix of 27 different salt mixtures was defined through the statistical design of experiments and using the "Full-Factorial" method (equation 3^{n-1} , where n is the number of salt combinations). In table 1 the upper and the lower limits of chemical composition using the SDE method is presented. Table 2 shows the matrix of the planned mixtures, in which the amount of NaCl is kept constant and the values of MgCl₂, CaF₂ and KCl vary.



Table 1: Range of salt chemical composition using the full-factorial method based on a commercial salt Flux 5

Industrial "Flux 5" (benchmark)		upper/lower limits around the industrial composition		
		-1	0	1
MgCl ₂	46	30	43	56
CaF ₂	5	0	5	10
KCl	23	15	23	31
NaCl	26	Constant (100-Σ)		

Table 2: Matrix of different salt mixtures prepared for the coagulation investigation

Nr.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	
MgCl ₂	X1	30	30	30	30	30	30	30	30	30	56	56	56	56	56	56	56	56	56	43	43	43	43	43	43	43	43	43	43
KCl	X2	15	31	23	23	23	31	15	31	15	15	31	23	23	23	31	15	31	15	15	31	23	23	23	31	15	31	15	
CaF ₂	X3	0	0	0	5	10	10	5	5	10	0	0	0	5	10	10	5	5	10	0	0	0	5	10	10	5	5	10	
NaCl	X4	55	39	47	42	37	29	50	34	45	29	13	21	16	11	3	24	8	19	42	26	34	29	24	16	37	21	32	

The salt mixtures mentioned in table 2 were investigated with respect to liquidus temperature via FactSageTM thermochemical calculations as well as Differential Thermal Analysing method (DTA) (one example see Fig. 1). These measurements showed that the salt mixtures 1 to 3, 10-22 and 25-26 shall have acceptable melting points between 374-716°C. The other mixtures with higher melting temperatures can never be used in a magnesium process. Therefore, totally 18 mixtures were selected from this matrix for experiments. On the other hand, the initially planned three different fluoride contents (0, 5 and 10 wt %) are not sufficient for consideration the role of fluorine-containing components. Hence, the number of steps for CaF₂ was increased to investigate the effect of fluorine clearly; however, the salt numbers were kept in the original numbers. In "new" salt mixtures, the value of CaF₂ was changed to 0, 1, 2, 3.5, 5, 6.5, 8 and 10 Wt %. The modified mixture matrix is represented in table 3, in which those salts with changed CaF₂ Wt % have been renamed with an "m-index".

Additionally fluor spar was replaced by other fluoride components such as KF, NaF, LiF and MgF₂ in "new" salt Nr. 19 (with 2 Wt % CaF₂) in order to investigate the influence of the type of fluorine-bearing components on coalescence efficiency.

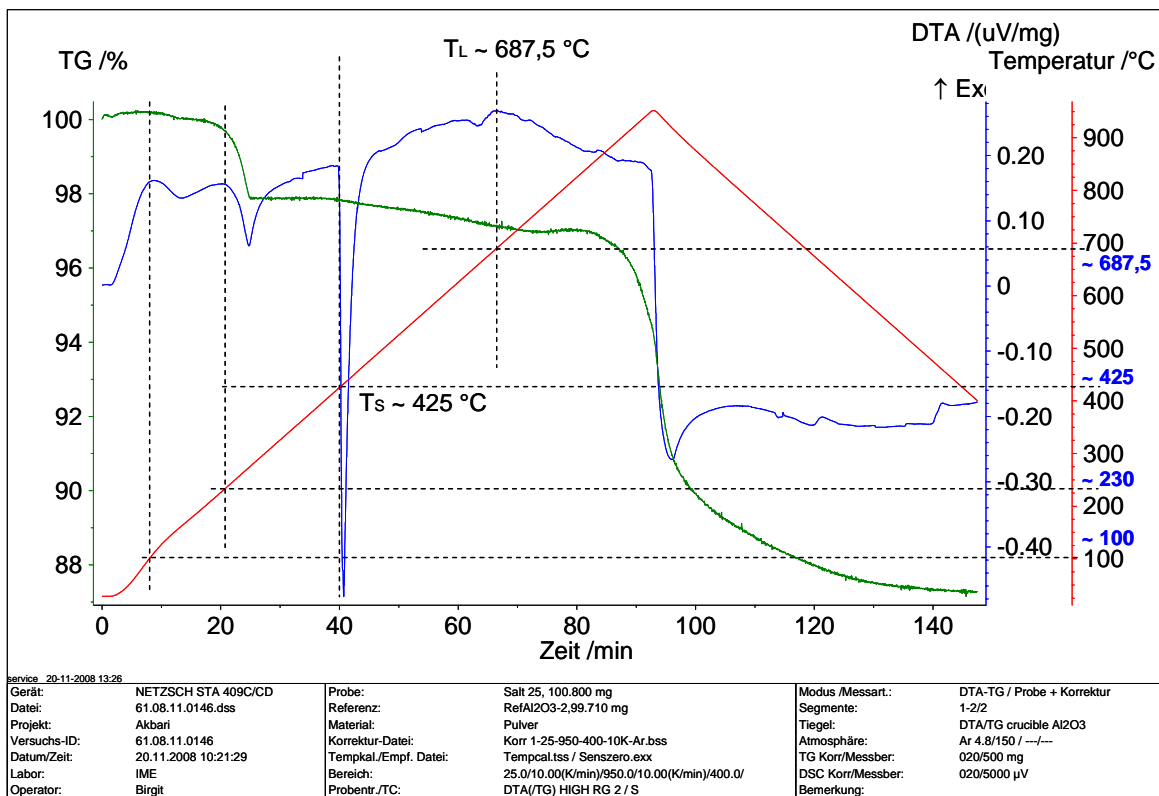


Figure 1: Exemplary DTA analysis (salt Nr. 25), showing a liquidus temperature of about 687°C, a solidus temperature of about 425°C, the evaporated moisture at 100°C as well as partial deterioration of crystallisation water at approx. 230°C

Table 3: Modified matrix of salt mixtures applied for experiments

Salt Nr.	1	3m	10	11	14	16	17	18	19m	20m	21m	22m	25
MgCl ₂	30	30	56	56	56	56	56	56	43	42.8	42.8	43	43
KCL	15	23	15	31	23	15	31	15	15	30.8	22.9	23	15
CaF ₂	0	1	0	0	10	5	5	10	2	3.5	6.5	8	5
NaCl	55	46	29	13	11	24	8	19	40	22.9	27.8	26	37

4 Coalescence Measurement Experimental Procedure

Compared to aluminium coalescence methods, where mostly pieces of aluminium foils are added to the molten salt bath, this process is much more complicated in case of magnesium. On the one hand, magnesium in the form of foil will burn tremendously directly on the surface if it is charged



into the molten salt. On the other hand, no standard method for magnesium coalescence has ever been published. Therefore a new coagulation methodology has been developed in this work, based on using magnesium chippings covered with powdery salt flux in a closed crucible. This method avoids the use of magnesium thin sheets as well as their contact with oxygen at testing temperatures.

Figure 2 illustrates four different chipping sizes drilled from a block of AZ31 magnesium alloy. For each coalescence experiment seven grams of magnesium chippings were applied, whose common size distribution is given in figure 3. In order to see the effect of each size on coalescence efficiency and to select a size for the main series, pre-tests were carried out using salt number 11.



Figure 2: Different chipping sizes distributed in magnesium chippings

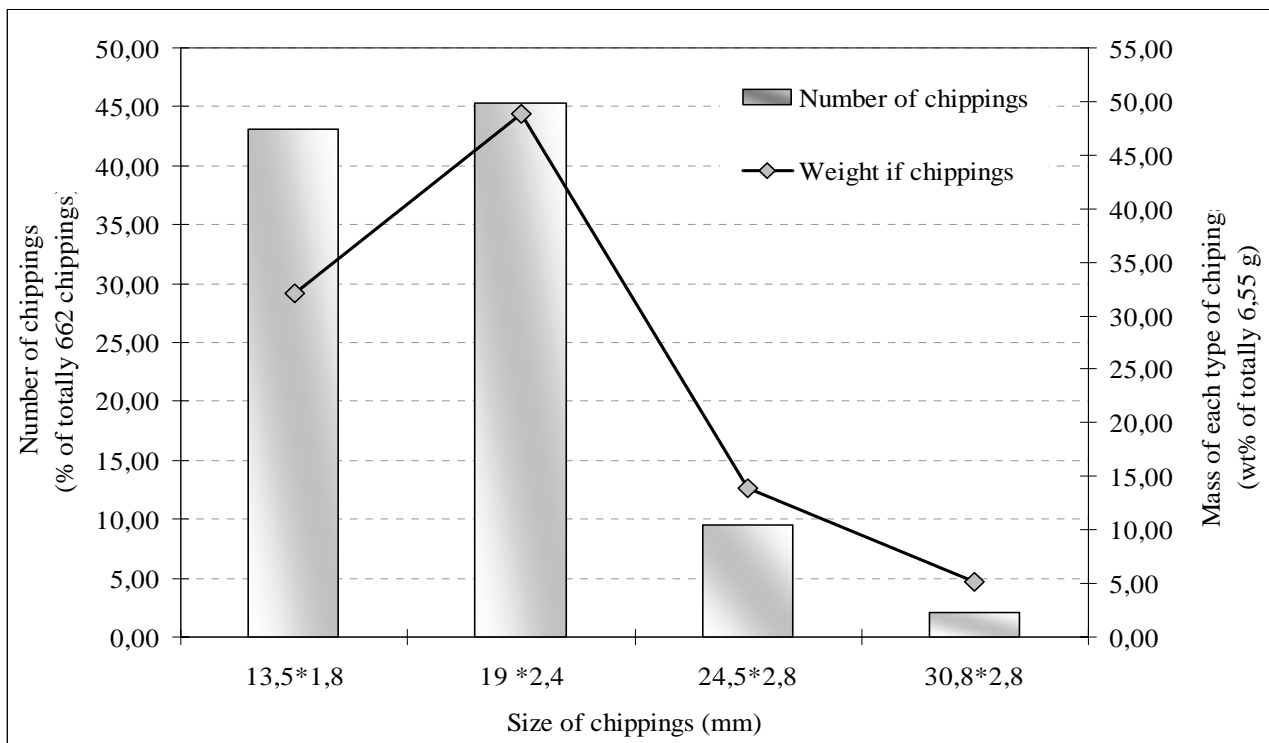


Figure 3: Distribution of chipping sizes, numbers and weight in approx. seven grams of chippings



In the experiments best effort was taken to charge identical magnesium scrap with respect to size, morphology and weight for coalescence experiments; therefore, such scraps were fabricated artificially. Before each individual test, magnesium chippings were inserted in boiling water for 10 minutes to be cleaned from the contaminations and to form oxide/hydroxide surface layers, then dried and held in a furnace at 200°C for 16 hours in order to strengthen the surface layers as well as to boost the oxidation/aging process. After this procedure, the chippings were ready to be used directly in coagulation experiments. Figure 4 illustrates the SEM/EDX analysis of chippings after the above mentioned scraping process, containing a thin layer of oxide on its surface.

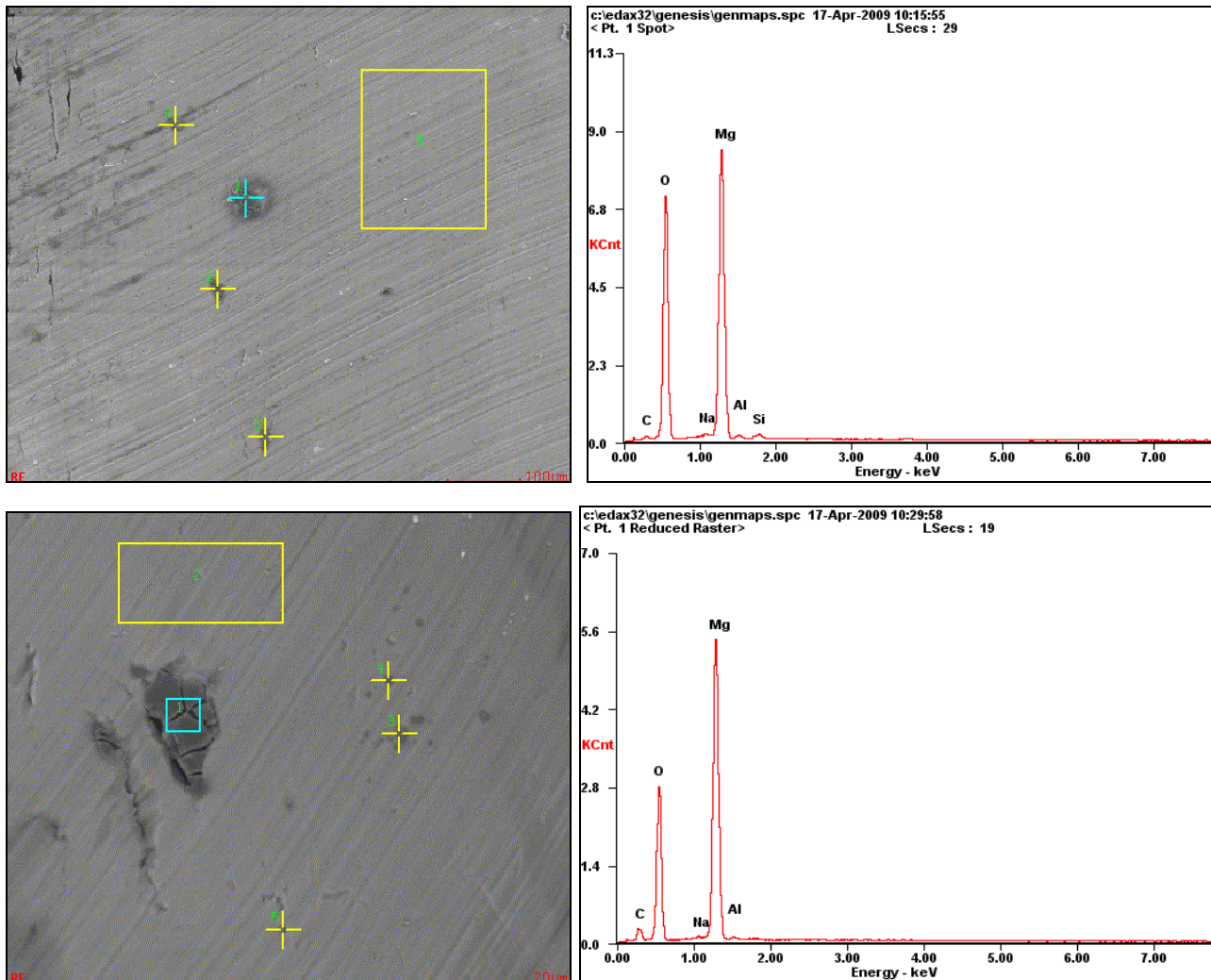


Figure 4: Exemplary SEM pictures as well as EDX- patterns of Mg- chippings after an artificial aging procedure showing a small amount of oxygen content (for both, the dark points identified with blue colour)

The all coagulation experiments took place in a small graphite crucible and in a resistance furnace at a constant temperature of 750°C. This temperature is approx. 100°C higher than melting temperature of magnesium as well as approx. 50°C higher than the highest salt liquidus temperature. Firstly



each of different salt mixtures shown in table 3 was prepared and weighed to obtain four times higher volume as Mg-chippings in order to assure that all chipping pieces have been surrounded by molten salt bath completely (see Fig. 5). Different holding durations were investigated to assess the most influencing retention time on the coalescence procedure. After holding at test temperature, the crucible was put into a bucket of copper chippings to be cooled down quickly. Afterwards, the solidified material was taken out from the crucible, crushed, sieved and classified into five different portions; the particles/balls with the diameter bigger than 7 mm, >1.6 mm, >0.8 mm, >0.4 mm and the rest.



Figure 5: Graphite crucible inside furnace containing magnesium chippings surrounded by two layers of salt mixture and covered up with a refractory lid

Generally speaking, the coagulation was calculated based on the particles bigger than 7 mm diameter. That means for those salt mixtures without any coagulated particles as big as or bigger than 7 mm, the coalescence efficiency has been taken as zero. For all other salt mixtures, the coagulation efficiency was calculated as “(weight of $\geq 7\text{mm}$)*100/ (total weight of deployed Mg-chippings)”.

5 Results and Discussions

5.1 Selection of the Chipping Sizes



Pre-tests showed that coagulation occurs mostly with smaller chippings (Fig. 6). That could be due to the interfacial tension effects, which need to be much larger for bigger chippings involving the stronger binding between molten salt and magnesium droplets, so they do not coagulate effectively. Hence, two of the smaller types of chippings were applied in the experiments.

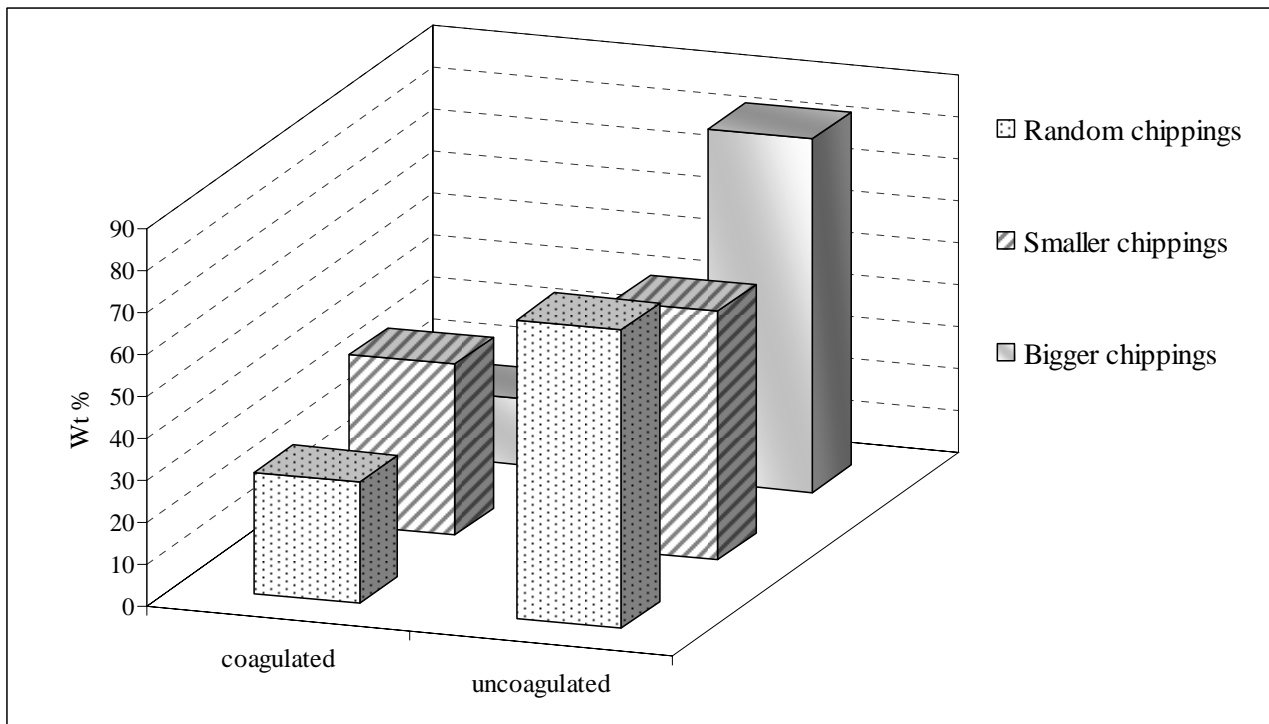


Figure 6: Effect of chipping size on coalescence efficiency (weight %) investigated through pre-tests

5.2 The Effect of Contact Period between Salt and Metal

Figure 7 illustrates that coalescence efficiency is being generally increased when the holding time rises from 0.5 to 1h, decreases at retention times of 1.5h and then staying quite constant up to 3.5h. It is supposed that coalescence efficiency is a function of time depending on various additives present in salt flux. Within an increase of contact time from 0.5 to 1h, the degradation of oxide film from the surface of metallic magnesium droplets proceeds significantly. However, higher holding times (1.5h) increases the risk of reoxidation of coagulated particles; hence, the efficiency decreases. The metallic loss is not possible to calculate, because the material fraction passed through the sieve with 0.4 mm (chapter 4) is mostly salt but also containing some amount of fine magnesium, which can not be separated. Unfortunately and contrary to aluminium coalescence trials, this material can not be washed and the exact value of metallic losses stays unknown. According to



these experiments, 1h was taken as the most optimum treatment time and used for all coalescence investigation.

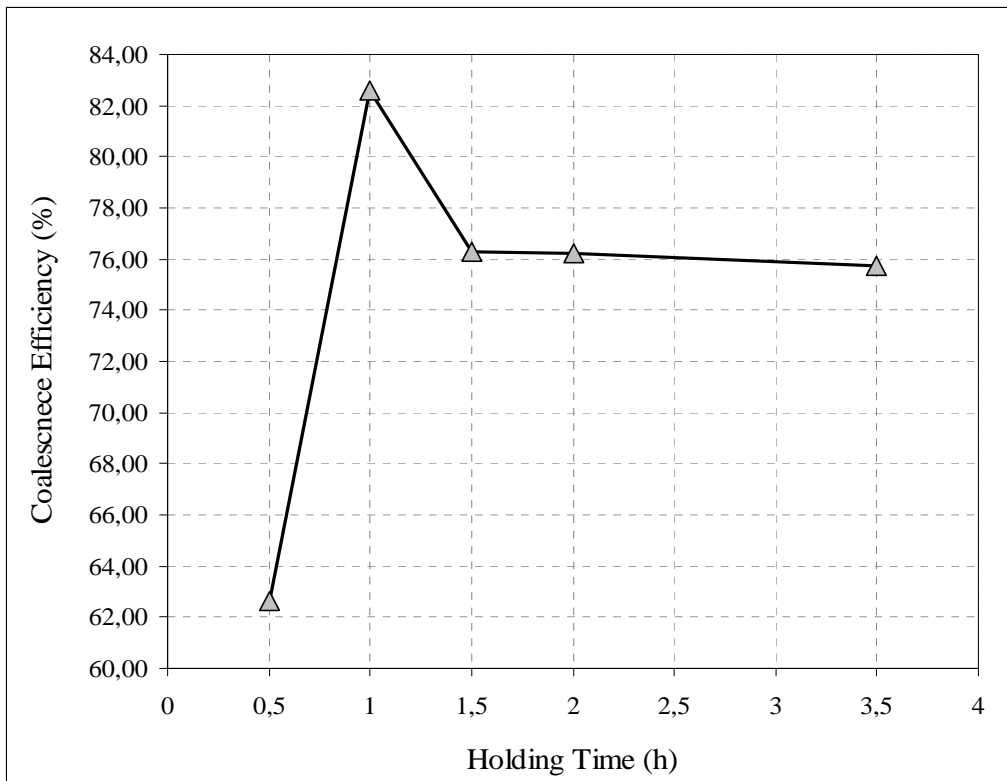


Figure 7: Coalescence efficiency at different contact times, in which salt mixture Nr. 16 was used together with Mg-turnings

5.3 The Effect of Chemical Composition of Salt Flux

Figures 8-13 illustrate the coagulation results of melting magnesium chippings with different salt mixtures mentioned in table 3 using the procedure described in chapter 4. Salt Nr.1 containing no CaF_2 and relatively little MgCl_2 showed the worse coagulation efficiency (Fig. 8). Through addition of 3.5 Wt % CaF_2 into the salt flux the coalescence efficiency was increased dramatically to more than 80% (see Fig. 9) showing the powerful influence of fluorspar in coagulation procedure.

In the absence of CaF_2 , the low coagulation efficiency could not be increased when more MgCl_2 was added to the mixture (e.g. in the case of salt Nr. 10), and if both MgCl_2 and KCl (e.g. the case of salt Nr. 11 in Fig. 10) are added, the efficiency increased only up to 20 %. It follows that without CaF_2 there is generally no chance for magnesium droplets to be coagulated; however, not only fluorspar but also the presence of chloride-containing components such as MgCl_2 and KCl is essential for the coagulation procedure.



Figure 8: Low-efficiency coagulation of Mg-chippings through salt mixture Nr. 1

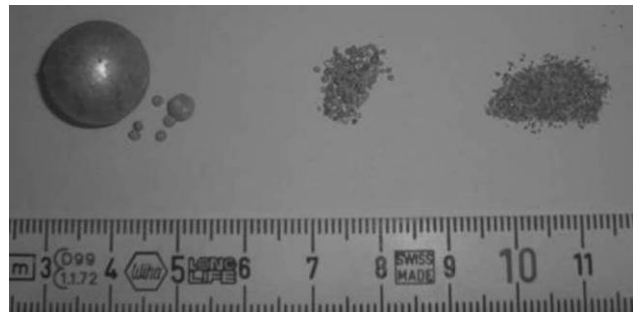


Figure 9: Efficiency of about 85 % after adding 3.5 Wt % fluorspar (salt mixture Nr. 20)

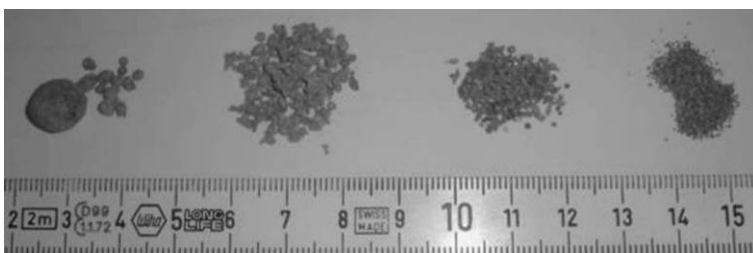


Figure 10: Only minor coagulation yield after addition of $MgCl_2$ and KCl without fluorspar in the salt mixture (Nr. 11)

Figure 11 represents the influence of different quantities of fluorspar on coalescence efficiency of magnesium droplets. Its value rises significantly by addition of CaF_2 up to 5 Wt %. This point, where three salt fluxes (Nr. 16, 17 and 25) with same CaF_2 but various amounts of $MgCl_2$ and KCl show quite similar effectiveness on coagulation, could be named as the peak of this curve (see also resulting droplets in Fig. 12). At further addition of fluorspar an unexpected descent in coagulation was observed, which can not be explained up to now (see also Fig. 13). On the other hand, Tenoroi and Delgado [13] showed also similar unexpected effects in the case of 10 Wt % NaF additions to $NaCl$ - KCl salt flux in aluminium coalescence. Through addition of more fluorspar, the coalescence rises again but still without achieving the maximum peak. It is supposed that 5 Wt % fluorspar has an effect like “over saturation” and adding more CaF_2 causes some non-dissolved particles on the surface of magnesium droplets, which avoid that these droplets coagulate together. Hence, the coalescence behaviour of salt fluxes by adding of more than 5 Wt % CaF_2 becomes worse.

In presence of 10 Wt % CaF_2 for two salt fluxes Nr. 14 and 18 (both including 56 Wt % $MgCl_2$) the coagulation of magnesium droplets has been quite similar, which could prove that in presence of highly enough fluorspar and magnesium chloride, the KCl doesn't play probably any big role in coagulation procedure.

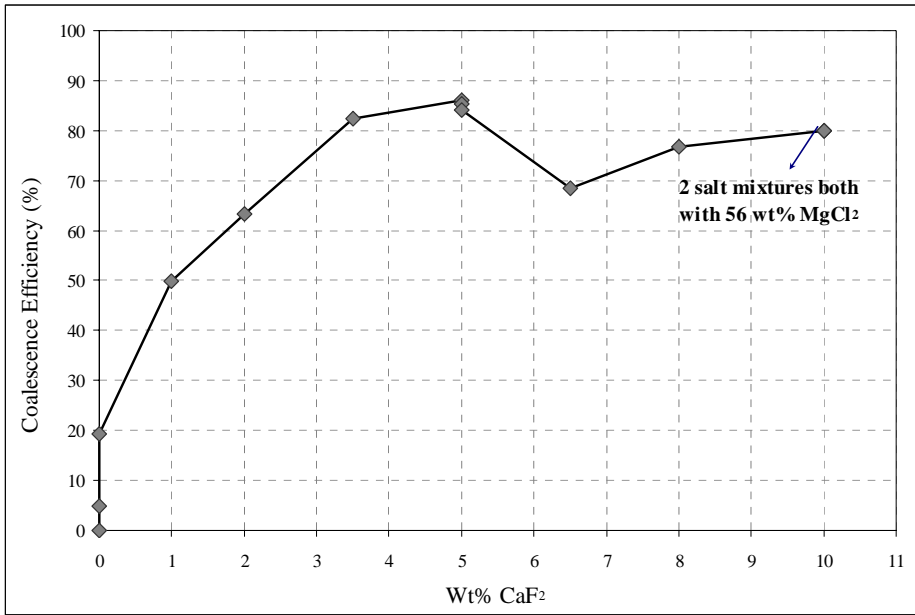


Figure 11: Coalescence versus the amount of CaF₂ present in the salt fluxes

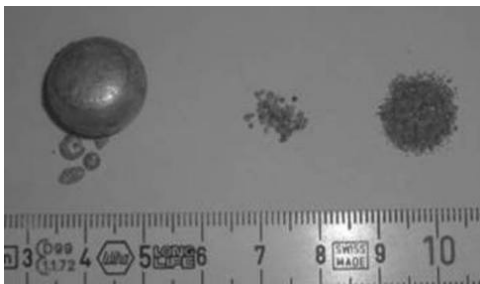


Figure 12: The best coalescence efficiency with approx. 86% achieved

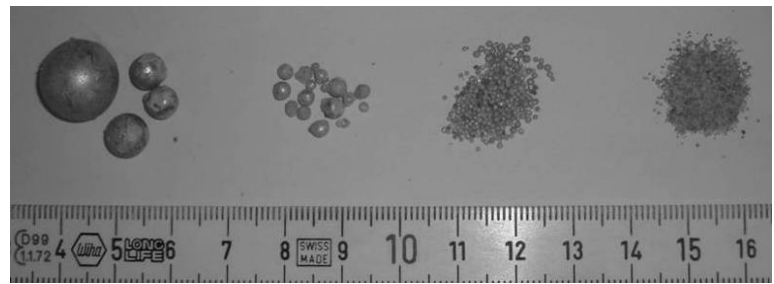


Figure 13: Further increase of fluorspar leads to reduced coalescence (salt Nr. 21)

In spider diagrams of figure 14 the complex relation between KCl and MgCl₂ in salt fluxes and coalescence efficiency has been illustrated. From the conducted series of experiments it can be supposed that coagulation of Mg droplets is rather independent from the quantity of KCl alone. This fact can be observed from the right side diagram in figure 14, in which the value of KCl changes without any regularity and the coalescence efficiency (CE) does not show any relation with these changes. In the case of MgCl₂ such a relation can be easier found for the salt fluxes Nr. 1 and 10, where more coagulation was achieved through higher additions. However, after consideration of salts Nr. 11 and 3, this relation is - in contrast - downward. On the other hand, fluxes Nr. 14 and 18 have the same quantity of MgCl₂ as fluxes Nr. 16 and 17; though, the last two ones caused more coagulation than the two formers.

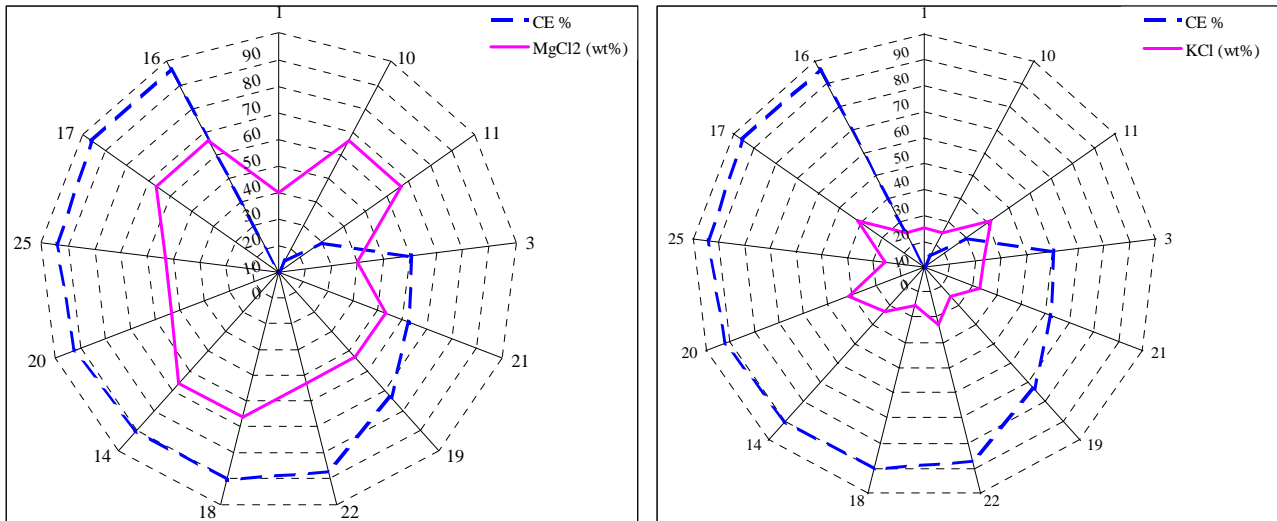


Figure 14: Coalescence efficiency of magnesium chippings in different salt mixtures (according to table 3), (left) the effect of $MgCl_2$ and (right) of KCl on coalescence

As a conclusion, neither $MgCl_2$ nor KCl alone has a defined influence on coalescence in comparison with CaF_2 , whose increasing effect is obvious e.g. from Fig. 11. However, it doesn't mean that the chloride-bearing components can not improve the coagulation behaviour of salt fluxes in magnesium processes, but it is clear here that the presence of fluorspar is definitely essential if an optimum coagulation is desired. Figure 15 correlates the major component concentrations of the selected salt fluxes on coalescence efficiency of magnesium droplets. This spider diagram again shows that fluxes containing 5 Wt % CaF_2 has the highest coagulation effectiveness. Also for fluxes with a high ratio of $MgCl_2/(KCl+NaCl)$ acceptable coagulation values can be reached, which prove again - as discussed before - the increasing effect of $MgCl_2$. From the resulted data it cannot be stated that the other salt components (KCl and $NaCl$) should be minimized to increase the coalescence, as according to flux 25 results this ratio is quite small, but the coagulation efficiency stayed in the highest level. Therefore, in the presence of 5 Wt % fluorspar (as the most effective concentration), the influence of all other salt components seem to be very small; although, their presence is necessary to maintain a low viscous melt, a matrix for non-metallic inclusion collection as well as for avoiding Mg -losses by chemical reaction. Such their amount shouldn't be too little.

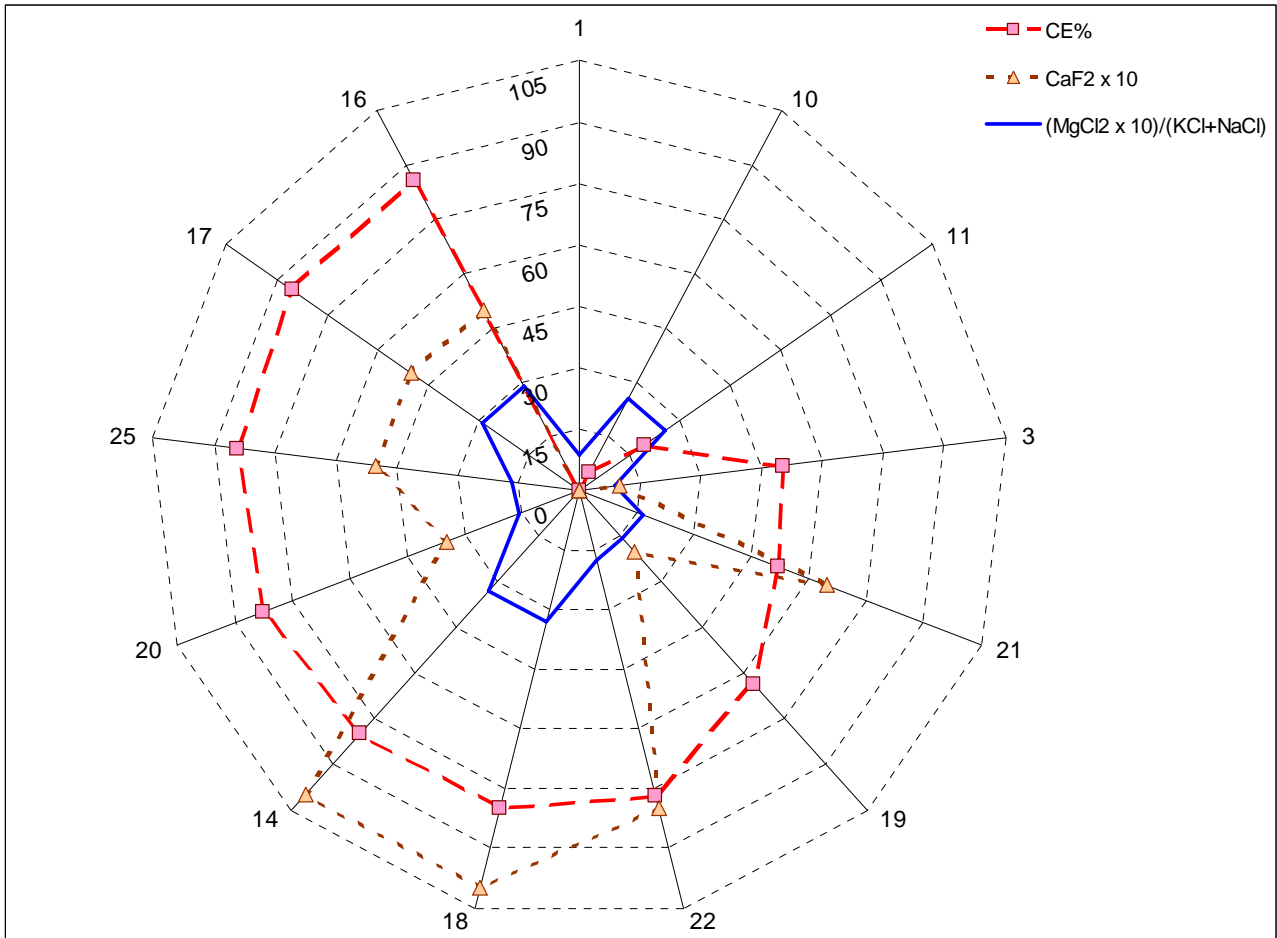


Figure 15: Coalescence efficiency of magnesium chippings in different salt mixtures (according to table 3)

5.4 The Effect of different Fluorine Compounds

The influence of the used fluorine compound in the flux is represented in figures 16 and 17. Lithium fluoride seems to have the biggest effect on coagulation of magnesium droplets. The coalescence behaviour was also improved if CaF₂ is replaced by KF. In the case of NaF addition no significant changes were observed comparable to CaF₂. The magnesium fluoride has the worse coagulation effectiveness.

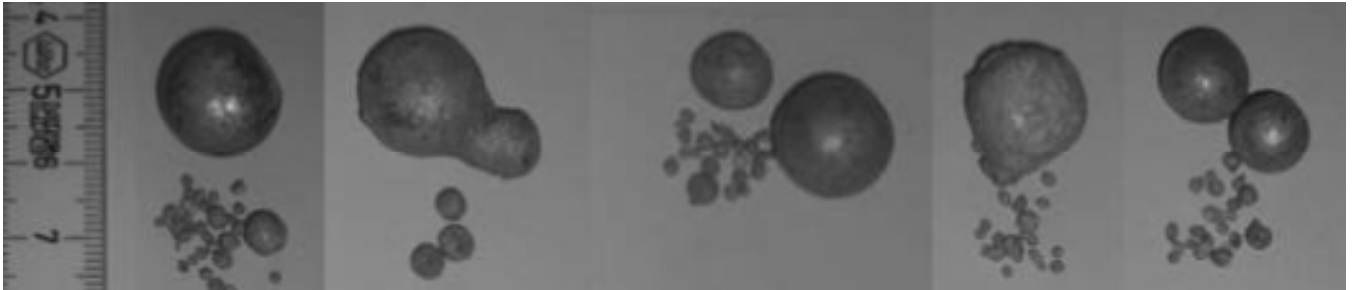


Figure 16: Coagulated droplets using different fluorine-containing components; from left to right LiF, KF, CaF₂, NaF and MgF₂ based on salt composition 19 (replacing 2 Wt % Fluorspar)

Sydykov [12] reported about fluorides effectiveness on Al-coagulation using a so called active fluorine ion content taking into account the F-content of each salt as well as the dissociation degree. If the completed dissociation reported by Sydykov is considered and accepted for this case too, the word “ion” can be hence used for fluorine. Whether also in the Magnesium system the value of fluorine ion present in a salt flux defines the effectivity of that flux is not clear up to now and is still under investigation. However, it can be easily seen from the following atomic weight series of the above mentioned fluorine components that the ratio of F-ion in each of these components does not follow any expected order represented in Fig. 17. That means, in spite of bigger amount of F-ion in MgF₂ than Calcium-, Sodium- and Potassium fluoride, the coagulation efficiency of magnesium droplets with this salt component is much less than that of the others.

LiF (73%) > MgF₂ (61.3%) > CaF₂ (48.7%) > NaF (45.2%) > KF (32.7%)

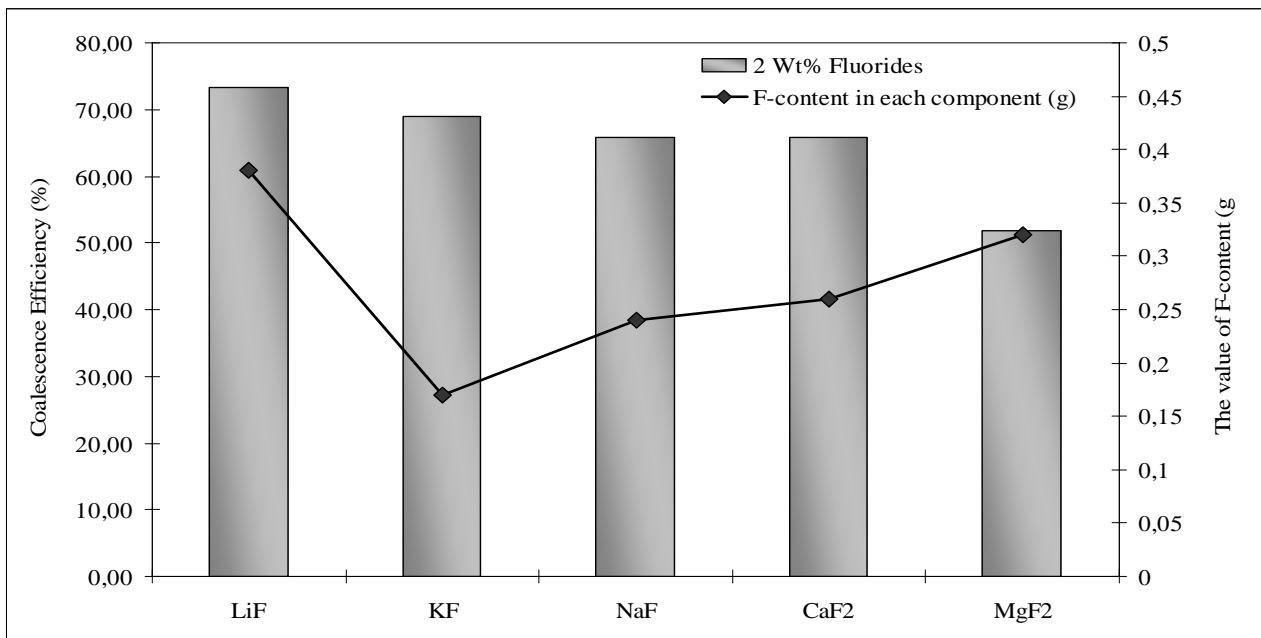


Figure 17: Coalescence efficiency of magnesium chippings using different fluoride-bearing components (all added by 2 Wt %)



That 2 Wt% of LiF addition leads to almost 1.5 times higher F-concentrations in the flux than 2 Wt % of CaF_2 . This suits to the experimental results of Fig. 17 too. Furthermore, the fact that NaF and CaF_2 have both quite similar contents of F-concentration and should have quite the same coagulation efficiency can be confirmed from experimental procedure too. But, the fact that KF has a less effectiveness on coagulation than NaF, CaF_2 and MgF_2 can not be proven by experimental trials. As a result, it is assumed that for Mg-system not only the quantity of fluorine ion in salt fluxes but also the presence of other elements such as Lithium, Sodium, Calcium or Potassium plays an important role in coalescence efficiency; therefore, it could not be concluded from these experiments that the more fluorine ion the more coagulation.

On the other hand, the coalescence trials were repeated for 5 Wt % of the above mentioned fluorides too and the results presented in figure 18 show that through increasing the value of fluorine (via addition of 2 and 5 Wt % fluorine-containing component), the coagulation of magnesium droplets has been increased too.

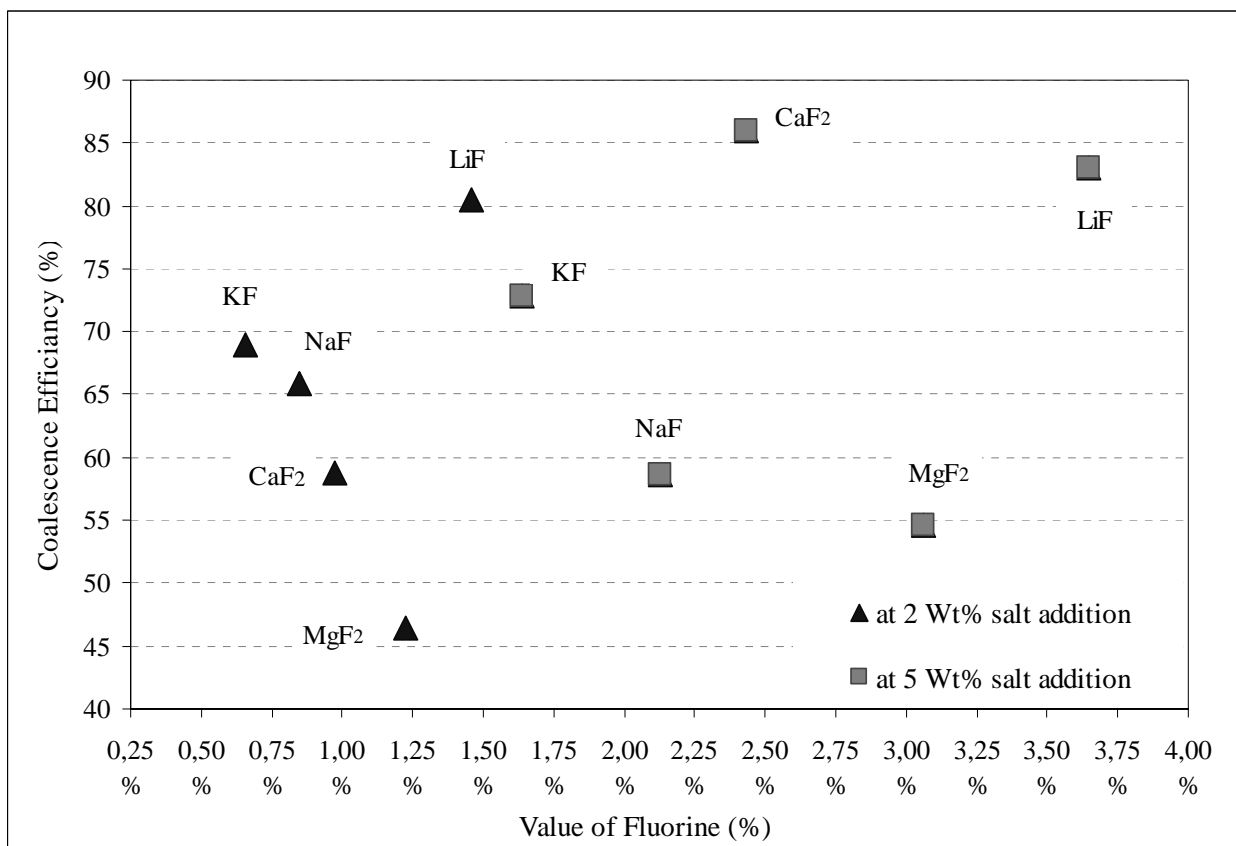


Figure 18: Coalescence efficiency of magnesium chippings versus the amount of fluorine

In the diagram of figure 18 and through an imaginary trendline, if an optimum coalescence efficiency of about 85% is desired, the value of F-ion after a complete dissociation of a fluorine-containing salt (without consideration the type of the salt) should be approx. 3.59 Wt %. According to actual salts price comparison (see Fig. 19), LiF and MgF_2 are anyway uneconomic choices to be



used in magnesium salt fluxes due to their extremely high prices. But, KF, NaF or CaF₂, whose prices are much less, could be good selections to be applied in magnesium salt fluxes. However, according to figure 18, the point related to 5 Wt % CaF₂ reaches a coalescence efficiency higher than 85%, despite having just 2.5 Wt % F-ion. This fact intensifies the above mentioned assumption of the importance of other elements such as Lithium, Calcium, Sodium or Potassium.

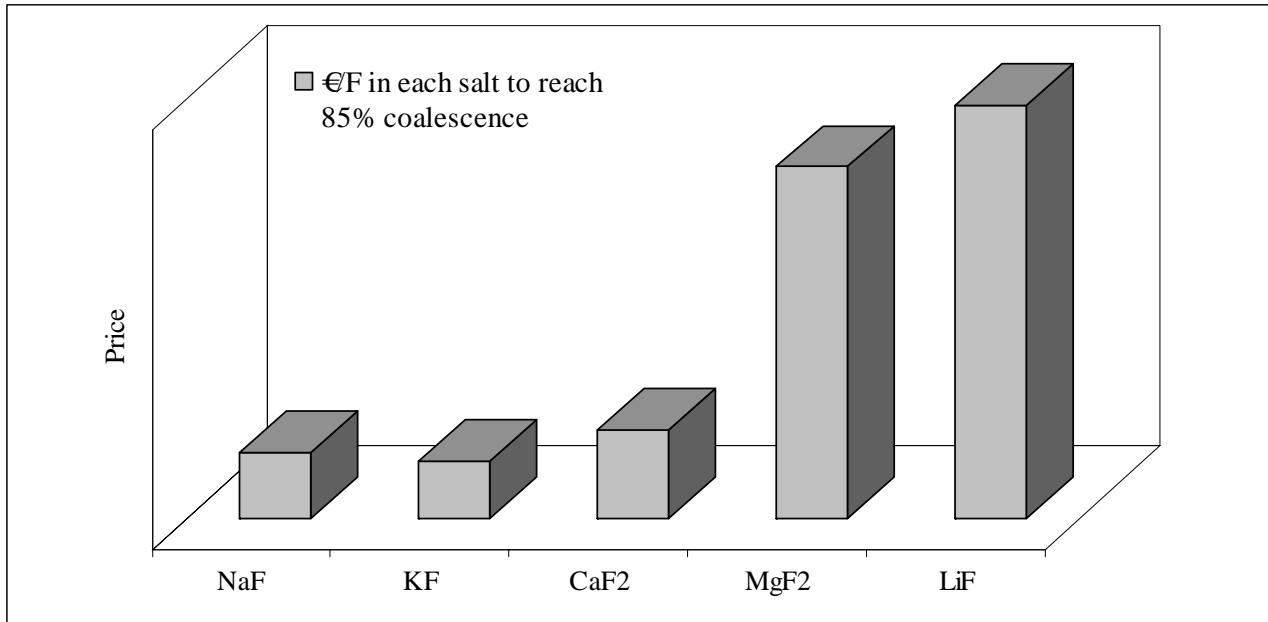


Figure 19: Comparison of prices for different salt compounds after statistics of Dec.08

6 Conclusion

In this paper the influence of the chemical composition of salt fluxes on coalescence of magnesium droplets was investigated. The results of this investigation are important to minimize the possibility of metallic magnesium losses during the recycling process. This loss, which is normally found as waste in the so called “black dross”, can be reduced if the magnesium droplets can be forced to coagulate, which leads to an improved separation from salt and oxide.

The experiments showed that a 5 Wt % of a CaF₂ addition (respectively 2.43% of F⁻) combined with 56 Wt % MgCl₂ in the flux will have the best effectivity on coagulation of magnesium drops, leading to approx. 86% efficiency. Furthermore, comparing different fluorine components, depending on the weight percentages of salt addition (2 or 5 Wt %) and also on the economical aspects, CaF₂ is still the best choice for applying in magnesium salt fluxes.

On the other hand, interfacial tensions between magnesium droplets, salt droplets and oxide particles play also an important role in the formation of black dross. For further studies, such investigations of interfacial tensions and wetting angles of the above mentioned materials are suggested.



7 Acknowledgement

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