Design of a Mini-TRF at IME – Methodology to Better Understand Salt Slag Interactions with Molten Aluminium

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Salt slags are complex by-products from aluminium recycling in TRF. Indeed, they are being modified all along recycling processes because one of their functions is to clean the metal and entrap impurities. The oxide content therefore increases during a recycling cycle and this changing composition influences the salt flux efficiency and leads to metal losses, by entrapment in the slag or by oxidation. This paper presents a new lab-scale furnace at the IME (Institute of Non-Ferrous Metallurgy and Recycling, RWTH Aachen University), the Mini TRF, built in order to better understand salt slag properties. This furnace has the characteristics to have a small scale and to be able to rotate and be tilted. The furnace dimensions enable to do fundamental research about mechanisms happening in slag while taking in account movement, as it is the case in industrial TRF.

Keywords:

Aluminium recycling – Tilting rotary furnace – Salt slag – Coalescence – Rheology

Aufbau eines Mini-kDTO am IME – Methodik zum besseren Verständnis der Wechselwirkungen von Salzschlacken mit flüssigem Aluminium

Salzschlacken sind komplexe Nebenprodukte des Aluminiumrecyclingprozesses im kippbaren Drehtrommelofen. Durch die Aufgabe der Salzschmelze, die Verunreinigungen der Metalle zu entfernen und diese aufzunehmen, wird die Salzschlacke über den Recyclingprozess modifiziert. Der oxidische Anteil steigt stetig an, wodurch die Eigenschaften der Schlacke sich ändern und die Effizienz der Verunreinigungsaufnahme beeinträchtigt wird. Folgen können Metallverluste durch z.B. dispergierte Metalltropfen in der Salzschlacke sein. Gegenstand dieses Artikels ist ein im IME (Institut für metallurgische Prozesstechnik und Metallrecycling der RWTH Aachen University) neu installierter lab-scale Mini-DTO. Der widerstandbeheizte Ofen ist sowohl dreh- als auch kippbar und soll ein besseres Verständnis der Schlackeneigenschaften ermöglichen. Der Ofenmaßstab ermöglicht Grundlagenforschung über die Mechanismen, die in der Schlacke von Bedeutung sind.

Schlüsselwörter:

Aluminiumrecycling – Kippdrehtrommelofen – Salzschlacke – Koaleszenz – Rheologie

Construction d'un Mini four rotatif inclinable à l'IME – Méthodologie pour une meilleure compréhension des interactions entre les scories salines et l'aluminium liquide

Construcción de un Mini-HRB en el IME – Metodología para comprender mejor los interacciones de las escorias salinas con aluminio líquido

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1 Introduction

Recycling is today an important part of the aluminium production, as the non-ferrous metallurgy has been largely developed in the last decades, and the primary production of aluminium needs 95 % more energy than the secondary production. One recycling possibility is taking place in Tilting Rotary Furnaces, with the use of a salt flux. The salt flux is mainly composed of the chlorides NaCl and KCl. The advantage of its use is that the metal bath surface is protected and not in contact with the air, which prohibits the oxidation of the metal, and the salt flux entraps the impurities like oxides and varnish residues. This cleaning operation leads to a constant evolution of the salt slag, as scraps are inserted in several times in the furnace. Therefore, the salt slag is a very complex component as its composition is changing during the whole process. At the end of the recycling process, the slag can contain up to 45 wt.-% oxides and 10 wt.-% metal.

This composition modification leads to a change of the slag properties: the salt flux becomes more and more viscous [1, 2] and the density difference between slag and aluminium will be always lower so that the metal/flux separation will be more complicated. Therefore, some metal droplets also stay entrapped in the salt slag. In order to reduce the metal content of the slag, some fluorides are added to the chlorides (often Na_3AlF_6 or CaF_2); this addition facilitates the cracking of the oxide layer on the metal droplets and enables their coalescence. This subject was largely considered by researchers, which is not the case of the movement influence on metal losses in TRF. Indeed, movement facilitates the cracking of oxide layers on metal droplets and promotes the meeting of metal droplets in the slag. This theme is often excluded from research and the IME decided to focus on it and to continue the fundamental research about metal losses in TRF. For this purpose, the IME has built a Mini TRF in order to be as close as possible to the industrial reality at a small scale.

2 Research done at IME until today

2.1 Coalescence tests at a small scale in a resistance furnace

The coalescence phenomenon and the role of fluorides have been nearly studied at IME.

Coalescence experiments have been conducted by Sydykov [3] and Besson [4] at small scale (crucibles with a volume of 150 ml) in a static resistance furnace. The experiments consisted in adding some small aluminium scraps in a salt bath with a determined composition and observe which fluoride addition was promoting the coalescence (Figure 1).

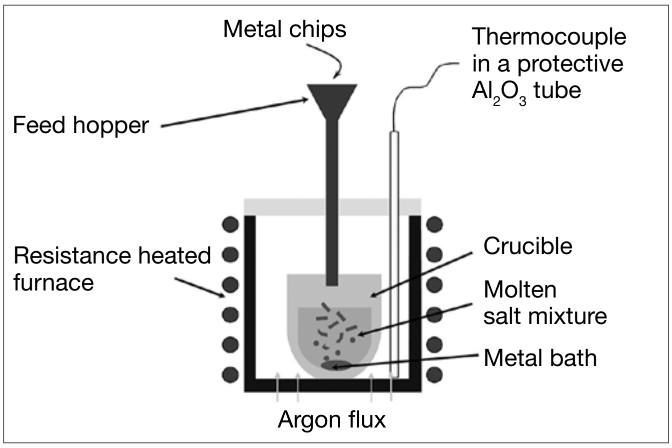

Fig. 1: Experimental set-up for the coalescence tests

The results analysis consisted in leaching the samples and sieving the metal droplets. The classification of the metal droplets by size conducted to the calculation of a coalescence criterion (Eq. 1), enabling to determine how much of the initial scraps coalesced. The coalescence is supported by the use of fluoride, for example cryolite. Coalescence efficiency was strongly dependant on the presence of Mg in the alloy (Figures 2 and 3) [4].

$$CE = \frac{\text{weight} > 2.5 \text{ mm}}{\text{total weight of recovered balls}} \cdot 100 \qquad (1)$$

The main advantage of these experiments is their scale. Indeed, the small scale enables to search the influence of each parameter separately. Besides, the experiments are short and do not need a lot of material and personal, therefore they can be repeated in order to obtain reliable results. Finally, it is also possible to test different atmospheres in the furnace. There are multiple experiments possibilities in this furnace.

However, the large drawback of this furnace is that only static trials are possible while movement plays a crucial role on the metal losses in a TRF. Indeed, it breaks the oxide layer on the metallic aluminium and promotes the meeting of free metal droplets in the slag. Static experiments enable to develop the research about the role of fluorides in the metal recycling process, but a rotating furnace would enable the research about the influence of the movement on the slag formation and the metal loss. Finally, static experiments necessitate a fluid bath whereas a salt

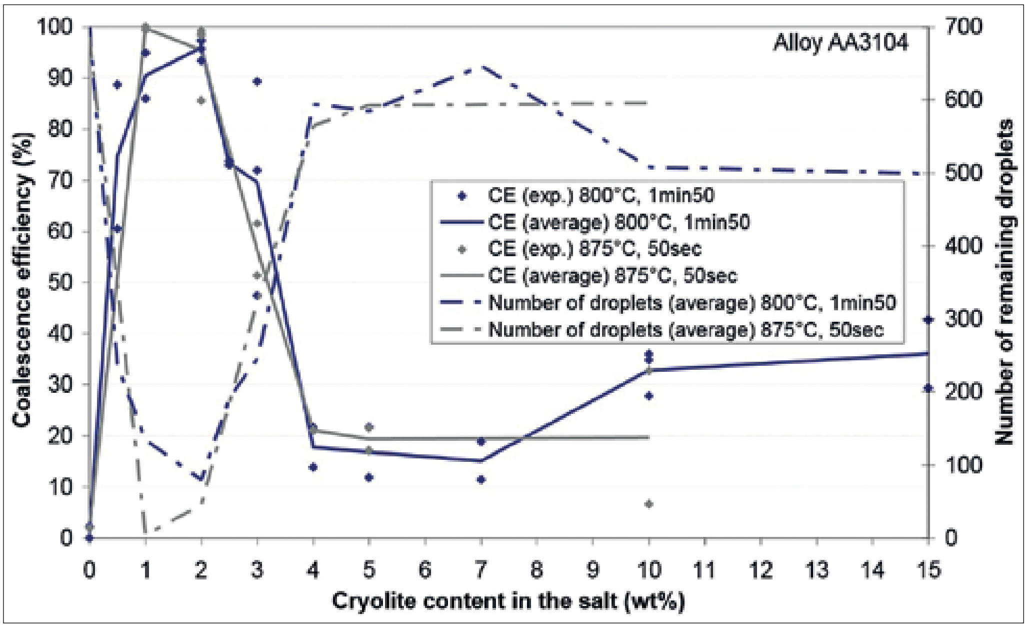

Fig. 2:
Coalescence efficiency of AA3104 (0.8 to 1.3 wt.% Mg) chips as a function of cryolite content in the salt (initial salt temperature = 800 °C or 875 °C)

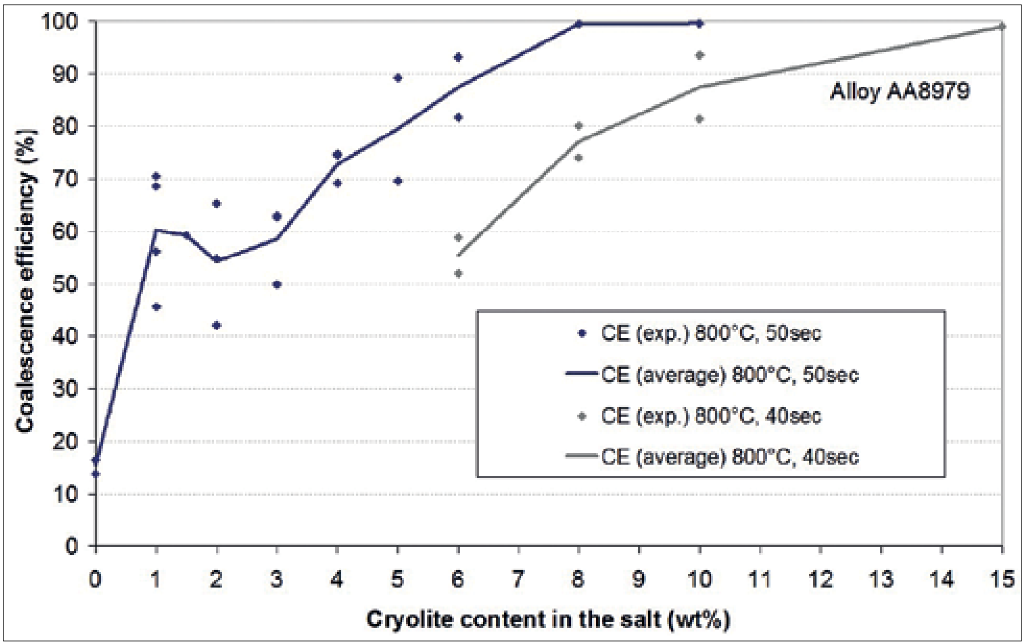

Fig. 3:
Coalescence efficiency of AA 8979 (low alloyed) chips as a function of cryolite content in the salt (initial salt temperature = 800 °C)

flux becomes drier during a recycling process because of impurities entrapment. This also leads to large changes in the flux properties, like the viscosity [1] which has a large influence on the metal losses in it.

2.2 TRF at demonstration scale

The IME Aachen also owns a large TRF, with an inner diameter about 1.4 m and a depth of 1 m, which corresponds to a volume around 1 m^3 in a vertical position. By considering a tilting of 70° from the vertical position (corresponding to the tilting used by industries for aluminium recycling), the volume is limited at 0.6 m^3. This furnace is heated by a gas burner, which is an advantage compared to the lab scale furnace as this heating modus is closer to industrial reality (Figure 4).

It is therefore possible to do recycling processes with this furnace which are very similar to the reality. This equipment is however not adapted to fundamental research about aluminium recycling, as this furnace asks a large amount of material and human staff. Therefore, it is often complicated to repeat trials because of a lack of time and material. Finally, the scale is so large that it is difficult to separate the different parameters and only study one phenomenon.

Fig. 4: Demonstration TRF at IME

2.3 Need of a small scale research equipment

In order to continue the fundamental research about salt flux properties and metal losses in aluminium recycling, a new furnace at small scale is needed. It has to be able to rotate and tilt, so the movement parameter can be taken in account in the research as it has a large impact on aluminium recycling in the industry.

The size of the furnace is also an important criterion, as repeating experiments will be more feasible with a small volume. Besides, a reduced volume enables to study parameters' influences separately. Finally, designing a new dynamic furnace with a scale comparable to the one of the lab-scale static furnace at IME would enable to determine the influence of movement on salt slag properties and functions.

3 Design of the Mini TRF

The Mini TRF is a small resistance furnace with an inner volume of about 0.3 l, so that the volume is comparable to other lab-scale static furnace at IME. This furnace consists in a stainless steel frame (cubic box) containing a graphite crucible, heated by electrical resistances and thermally isolated. The pattern of the furnace can be seen in Figure 5. The furnace is closed by a lid which can be opened at any time with a wooden handle.

The furnace has two specific properties. First the stainless steel frame can be continuously tilted from 0° (vertical position) to 65°. The furnace in a tilted position can be seen in the left photo of the Figure 6. Besides, the inner graphite crucible rotates from 0 to 40 rpm. The rotation of the axis is carried out by an electrical motor.

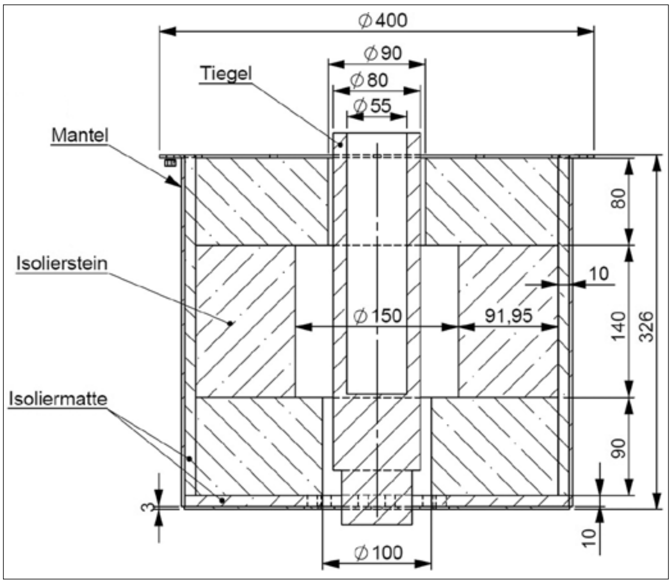

Fig. 5: Pattern of the Mini TRF (lengths in mm)

Fig. 6: Photos of the Mini TRF (left: overview, right: zoom on the opening)

The movement of the furnace does not interrupt or disturb the heating process of the furnace. A temperature of 1200 °C can be reached inside the graphite crucible.

The furnace has an electrical control system, where temperature profiles can be programmed, and rotational speed and tilting angle controlled. The Mini TRF works under an inert gas, argon. The gas inlet pipe is situated on the lid. In the lid is another hole, where thermocouple and gas measurement systems can be inserted (Figure 6, right photo).

As the graphite crucible is hardly removable from the furnace at high temperature, trials are done in alumina crucibles to facilitate the experimental set up. These alumina crucibles are inserted in the graphite outer crucible.

4 Commissioning of the furnace with first results

4.1 Furnace parameters

In order to validate the furnace, recycling cycles at a very small scale were conducted and the final products were analyzed. The experimental set up was done so that the conditions are as close as possible to the industrial reality. The parameters range of an industrial furnace and those chosen for the Mini TRF for the validation of the furnace are summarized in Table 1.

Table 1: Parameters of a standard industrial TRF and chosen for the validation of the Mini TRF

Furnace	Tilting angle [° from the vertical position]	Inner diameter [m]	Rotational speed, rpm [min^{-1}]
Industrial TRF	70-80	3-3.5	1-2
Mini TRF	65	0.06	30

The tilting angle chosen for the validation was chosen as close as possible to the value from a standard industrial furnace.

The rotational speed was calculated considering that the Froude number from an industrial TRF is equal to the one of the Mini TRF [5]:

$$N_{Fr}^2 = \frac{n^2 D \pi^2}{g}$$

with = Froude number, n = revolutions per second [s^{-1}], D = inner diameter [m], g = gravitational constant = 9.81 m·s^{-2}

Supposing that an industrial furnace has a rotational speed about 1 to 2 rpm and a diameter of 3 to 3.5 m, the rotational speed of the lab scale furnace should be adjusted to around 25 to 35 rpm in order to reach the same conditions. The mean value of 30 rpm was taken for conducting the experiments.

4.2 Input material

A scrap charge of UBC's (mini-bales) was chosen for the validation. As the Mini TRF is an electrical furnace and is not heated by a burner, it was chosen to do a preliminary separated pyrolysis of the bales to remove the varnishes. This pyrolysis was conducted during 1 hour at 500 °C in an electrical furnace where argon was injected. The pyrolysed input scraps are shown in Figure 7.

The flux which was used in the Mini TRF consisted of a recycled equimolar NaCl-KCl salt, with an addition of

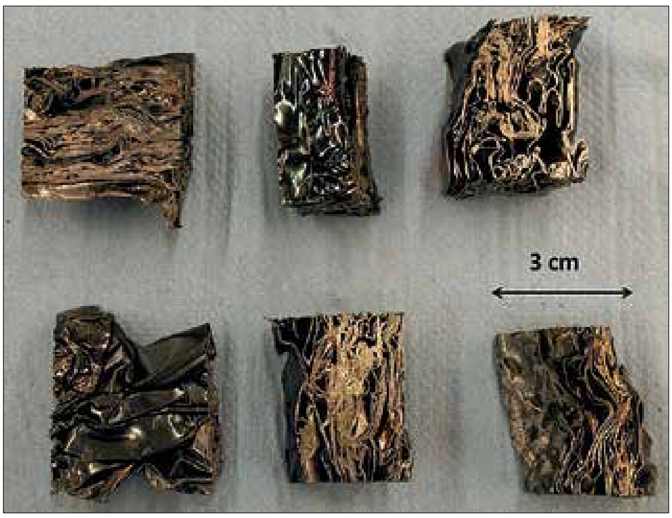

Fig. 7: Input material after pyrolysis

2 wt.-% cryolite. Different salt/scraps ratio were tested in order to determine which conditions lead to a final salt slag similar to industrial ones. The parameters of the different experiments are summarized in Table 2.

Table 2: Experimental input material

Salt/scraps ratio	Scraps	Salt
0	75 g of pyrolysed scraps (6 bales)	98 wt.-% of equimolar NaCl-KCl salt + 2 wt.-% Na_3AlF_6
1/3		
2/3		
1		

Simultaneously investigations of salt melts with small KCl proportions (5 to 30 wt.-%) were done. Pyrolysed UBC's (Figure 8) were melted in present of fluorides like CaF_2 and cryolite. Furthermore the influences of compact and loose input material were examined.

Fig. 8: Pyrolysed UBC chips at 550 °C, compact pyrolysed UBC chips

4.3 Description of the experimental set up

All experiments were performed at 750 °C.

In a first time, the salt was melted in the furnace. The 75 g of UBC's scraps were then inserted in the furnace in two times, with a waiting time of two minutes between the two charges. After the insertion of the last UBC's bale, the temperature of the furnace was maintained at 750 °C under an argon atmosphere during 15 minutes. After that time, the crucible was removed from the furnace and air cooled.

In the second test series the salt systems 70 wt.-% NaCl – 30 wt.-% KCl; 80 wt.-% NaCl – 20 wt.-% KCl; 95 wt.-% NaCl – 5 wt.-% KCl were examined. Two additives namely CaF_2 and cryolite were added in the molten salt bath. After dissolving of the fluorides UBC's were molten inside the bath. The angle and rotation rate of the Mini TRF were equal for all trials.

In order to separate the slag from the metal and analyze it, the samples were leached in distilled water in order to dissolve the salt. The solutions were then filtered with fine paper and the residue was dried at 150 °C. This residue was finally ground in order to separate the metal from the impurities. The grinded residue under 90 µm was analyzed by X-ray fluorescence and with the brome methanol method in order to determine the metallic content. The part above 90 µm was supposed to be only metallic. The solution underwent ICP analyses.

4.4 Analysis of preliminary results

A visualization of the different samples is presented in Figure 9. The obtained dross and metal and metal bath have different aspects, depending on the salt/scraps ratio. Without a salt usage, the obtained sample is very brittle and dirty; the impurities are mixed to the metal. By using salt, it can be easily noted by its light color that the metal bath is clean. An increase of salt leads to denser slag and reduces the metal entrapment in slag.

Two of the salt functions are brought forward with the metal recovery results in Figure 10. Indeed, the metal bath increases with the salt factor, which agrees with the facts that salt promotes the metal coalescence and cleans metal from impurities. The maximum metal bath is above 97 wt.-% of the initial charge, with an excess of salt, where no metal droplets could be found.

The experiments underline the salt factor significance as regards to the impurity degree of scraps: a reduced salt addition leads to a large metal content in the slag, which reaches 29 wt.-% of the initial charge for a salt/scraps ratio of 1/3 (Figure 11). The metal content values are of course not comparable to the industrial reality as the experiment duration was chosen so that differences between each trial are clear.

Fig. 9: Cooled samples after recycling cycles (up left: Salt/scraps = 0; up right: S/s = 1/3; bottom left: S/s = 2/3; bottom right: S/s = 1)

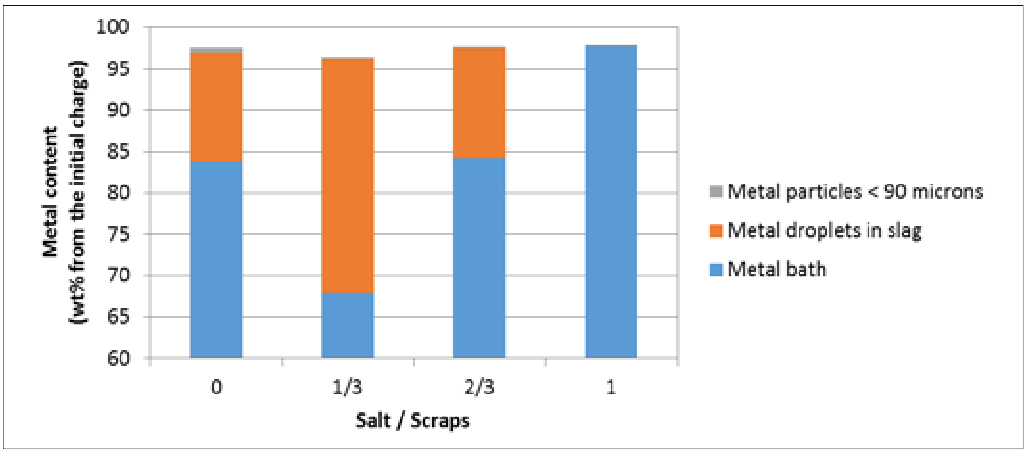

Fig. 10:
Metal recovery of the samples depending on the salt factor

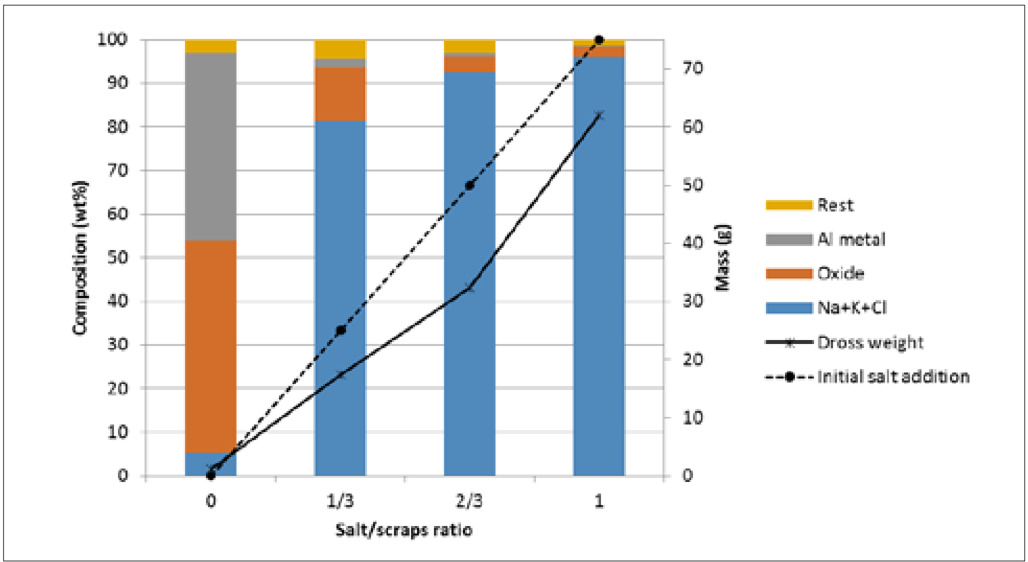

Fig. 11:
Dross/slag characteristics

In the case of the free-salt experiment, the metal bath is similar to the values obtained by a 2/3 salt factor. However, the metal quality is worse as pyrolysis residues are remaining.

Finally, the missing part of the initial charge corresponds to pyrolysis residues (rests of varnish and oxides), and the rest is due to oxidation in the furnace. Indeed, the argon atmosphere is briefly broken by opening the lid and loading the furnace.

The dross amount formed during the recycling processes strongly depends on the salt factor: it normally increases with the salt quantity initially inserted in the furnace. However, it can be observed that the final mass of slag is always lower than initial salt weight; this difference can reach 15 g and is due to the salt evaporation during the process. The free-salt process leads to a reduced amount of dross, lower than 2 g.

Salt is largely the main component of slags coming from processes with salt usage. Its content is already higher than 80 wt.-% for a salt factor of 1/3 going and increases until 96 wt.-% for the highest factor.

By calculating the oxide mass in dross, it appears that whatever the salt factor chosen for the recycling process, this mass stays constant, between 1 and 2 g. This constancy proves the airtightness of the Mini TRF; this oxide content consists of pyrolysis rests, coming from the initial charge and no or a negligible oxidation happens in the furnace. The rest of the slag mainly consists of fine metallic particles and carbon residues.

These compositions are far away from industrial black dross compositions from TRF, where the oxide content consists of 35 to 60 wt.-% of the slag [6, 7], depending on the type of charge, the salt factor and the burner settlings. The gap between the results and industrial slag compositions is due to the fact that a lower salt factor is often used and some oxidation may happen in an industrial TRF.

The test series with reduced KCl component show that cryolite supports coalescence regardless of whether the input material is loose or compact. The coalescence of the loose UBC chips is insufficient, if CaF_2 is added (Figure 12). The metal droplets are entrapped in the salt slag. Separation of salt slag and metal ingot couldn't be achieved. Indeed, compacting of the material induces better coalescence in the case of CaF_2.

These first results enable to see the evolution of the salt efficiency in relation to its factor and highlight two of the salt functions. The next step is to take the oxidation factor in account.

Fig. 12:
Crucible content of trials with 2 to 3 wt-.% CaF_2 (regardless of KCl content and salt/scrap-ratio)

5 Improvement of the furnace

The furnace presents very innovative possibilities and the first results are promising. However, some improvements are planned on the furnace in order to expand Mini TRF's abilities.

The largest limiting factor today is the atmosphere in the furnace. The Mini TRF can only work under an argon atmosphere. This possibility is interesting for analyzing phenomena other than oxidation, but it would be better to have the possibility to work under different atmospheres. Besides, the heat transfer through the actual graphite crucible is variable considering the graphite combustion. The realization of a ceramic crucible is currently in progress; this material is sustainable and will able to work with oxygen.

The loading process also has to be optimized, as it requires the complete opening of the lid which leads to an atmosphere change in the furnace. It is therefore planned to build a loading tube, which could be inserted directly in the lid of the Mini TRF. This would minimize the introduction of air into the furnace.

6 Summary

The Mini TRF is an innovative furnace enabling to go further in fundamental research about aluminium recycling with salt usage. Through its tilting possibility and its rotation, the Mini TRF has similar characteristics to industrial tilting rotary furnaces, while staying at a small scale. It therefore enables to separate the different salt properties and eases the understanding of the different phenomena.

Even if the furnace has been built for the theme aluminium recycling, the Mini TRF also presents opportunities for other non-ferrous metals like magnesium or copper because of its large panel of parameters.

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