Development of a Sampling Device for Furnace Dross

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Abstract

A series of tools for sampling dross generated by aluminium melting furnaces were developed and tested in furnaces at Sapa Heat Transfer, Sweden and Hydro Sunndalsøra, Norway. The goal was to create a device that is easy to use and gives samples representative of the dross inside the furnace. Metallic aluminium content was measured by re-melting the samples of the furnace dross with salt flux, separating the oxide and metal, and weighing the mass of the recovered metal. The key to a successful result was choosing a sampling device with the correct design to allow metal to drain as the sample was taken.

Introduction

Dross is generated on the surface of molten aluminium by contact with oxygen. Dross is a mixture of aluminium and its oxide and Non Metallic Product (NMP).

Oxidised aluminium is irreversibly lost. However, entrapped metal can be partly recovered at the furnace site or treated at another location, such as a dross processor.

Industrial aluminium dross is usually regarded as a heterogeneous mixture of large lumps of dross, fine oxides, and metal. Therefore, sampling and examining of a representative material is regarded as difficult. The metal content of the dross may depend on how skimming of the melt is executed, in addition to molten alloy composition, fluxing, and the dross-cooling process [1].

Several methods have been used to characterise dross and evaluate the metal content, as seen in Table I. The metal content was characterised by adding salt or acid, together with physical detections of density and size distribution.

In this project, a simple, fast and applicable method for the industry was sought, and therefore re-melting with salt was determined as the method for separating the aluminium from the oxide and NMP.

Argonne National Laboratory [2] developed a sampling procedure to collected dross from the furnaces. A clay crucible, nominally 10-15 cm in diameter at the surface and 10 cm deep, was held in place at the end of a long steel rod. The crucible was dipped sideways below the surface into the molten Al, rotated, and then scooped upward to collect both molten metal and the surface dross layer. The dross samples were allowed to cool inside the crucibles. After being cut in half and polished, the cross section is as shown in Figure 1.

| Table I Summarization of dross characterisation methods | | | | | | |
|--|---|--------|--|--|--|--|
| Characterisation | Method | Source | | | | |
| Bulk density of granular dross | By the weight of the dross put into a vessel of known volume | [3] | | | | |
| Apparent density of compact dross | By the volume of the pieces and their weight in liquid paraffin. | [3] | | | | |
| Particle size analysis | By sieving | [3] | | | | |
| Salt contents | Water leaching | [3] | | | | |
| Metal content | The dross was melted at 750°C with a mixture of 70% NaCl, 28% KCl, and 2% CaF_2 in a graphite crucible. | [3] | | | | |
| Metal content | Measure bulk density as above. Dross was broken into small pieces and chemically leached. Approx. 10 dm ³ of 6N HCl was slowly stirred for 2 hours into 5 kg sample. The non- soluble residue was collected on the filter after vacuum filtering, then being cleaned with distilled water and dried. | [4] | | | | |
| Metal content | Put dross sample into a bottle with 100 ml 2N HCl acid. | [5] | | | | |
| Metal content | $\begin{array}{llllllllllllllllllllllllllllllllllll$ | [6] | | | | |
| Morphological and compositional differences in compact dross | Extensive electron probe microanalysis (EPMA) Backscatter electron (BSE) imaging in the EPMA | [7] | | | | |
| Crystalline phase content in compact dross | XRD/TEM | [7] | | | | |



Figure 1 The cross section of the bulk dross sample taken by Argonne National Laboratory [2]

Aluminium is trapped in dross due to the oxide skin strength [10] or surface tension of the dross skin in the furnace [8]. Das

also saw under EPMA that the aluminium melt was trapped within the extensive network of oxide layers [2]. Besson and coworkers [9] have demonstrated that small addition (1 - 5%) of cryolite to an equimolar mixture of NaCl – KCl salt improves the coalescence of aluminium droplets during dross treatment. The interface tension between the salt and the molten aluminium is reduced. Minimizing the interfacial tension between salt and metal increases the affinity between metal and salt and eases stripping the oxide layer off.

Experimental Procedure

Dross sampling

The dross samples were taken with (i) sieve and (ii) cup samplers (shown in Figure 2) from (a) the melting/holding furnace and (b) in the dross pan after dross skimming, at a remelter Sapa Heat Transfer and from one furnace at primary aluminium producer, Hydro Sunndalsøra. Dross samples were taken from remelter using small-, medium-, and large-hole sieves.



Figure 2 Dross sampling devices: sieve (left) and cup (right)

The secondary metal was melted in the melting furnace, and then transferred to the holding furnace by a launder. The salt was added just before skimming and dross samples were taken from both furnaces and both dross pans as shown in Figure 3.



Figure 3 Dross sampling from the melting furnace (left) and the holding furnace at the remlter (right)

With one furnace operation at the primary producer, the metal was alloyed, stirred, and then skimmed. Dross samples were taken after stirring and before skimming in the furnace, and after skimming from the dross pan, as shown in Figure 4 and Figure 5. For one of the charges (P4) 0.5% Mg was added to the charge before stirring.

The dross samples were randomly divided into four parts. Up to three out of four parts of each dross sample were re-melted, indicated by the three green bars at P4_DP in Figure 8.

Table II gives the charge information. There are some differences in %Si, Cu, Mn and Mg between the charges. Since Mg was added before skimming in P4, the magnesium content of this alloy is significantly higher than the other three. At primary producer cold metal was added to the melting furnace in addition to the hot primary metal.



Figure 4 Dross sampling from the furnace at primary producer



Figure 5 Dross sampling from the dross pan at primary producer

Sample remelting procedure

The final procedure for separating aluminium out of the dross is as follows. An empty crucible¹ was heated to 1000°C in an induction furnace. When 1000°C, measured by a K type thermocouple, was reached the heating power was reduced to maintain the temperature of the crucible, and a sample of approximately 1 kg dross was added. When the dross reached 1000°C it was stirred with a steel rod. This process was repeated until the dross was melted. Then the same amount of salt was added to the crucible. When the mixture reached 1000°C it was stirred until the aluminium and salt was melted and well mixed, that is it felt like stirring water. Then the liquid was cast into three moulds (salt fraction, aluminium and salt, non-metallic product). The cast ingots were cooled for a few hours, and the weight was measured, before immersion in water overnight. All residues > 1 mm were sieved and metal pieces or droplets > 4mm were collected and weighed. The weight of metal and NMP was recorded. Numerous gas bubbles were observed on the ingots in the water. According to chemical analysis of the salt used in the remelting, is contained 2%Na₃AlF₆, 19 KCl, and 79%NaCl.

In the first trials of remelting Sapa dross, the dross was heated to 900°C. The salt was then less viscous and when pouring out, the liquid aluminium was poured into the mould, and the salt, some aluminium and NMP remained in the crucible. The left over in the crucibles were remelted again and poured out as the second

¹ Morgan foundary crucible with 30-50 wt% flake graphite. Morgan Advanced Materials, Quadrant, 55-57 High Street, Windsor, Berkshire, SL4 1LP, England.

metal fraction. Whatever aluminium metal that was left in the bottom salt and NMP was finally separated by hand. This was a

more time consuming method than using a temperature of 1000° C.

Table II The alloy composition in the furnaces at Hydro Sunndalsøra and Sapa.

P1 – P4 represent samples from four different charges at Hydro Aluminium, Sunndal Norway and R1 and R2 samples are from two different charges from SAPA Heat Transfer, Finspång Sweden.

| | | Prir | Remelt | | | |
|-------------|--------|--------|--------|--------|--------|--------|
| Test Number | P1 | P2 | P3 | P4 | R1 | R2 |
| Si [wt%] | 0.4152 | 0.4792 | 0.4045 | 0.4192 | 1.937 | 1.942 |
| Fe [wt%] | 0.1830 | 0.1969 | 0.1714 | 0.1933 | 0.4580 | 0.4512 |
| Cu [wt%] | 0.0010 | 0.0010 | 0.0008 | 0.001 | 0.2009 | 0.2033 |
| Mn [wt%] | 0.0209 | 0.0205 | 0.0197 | 0.0188 | 0.8880 | 0.8830 |
| Mg [wt%] | 0.030 | 0.061 | 0.090 | 0.4586 | 0.182 | 0.1702 |
| Zn [wt%] | 0.002 | 0.003 | 0.002 | 0.002 | 0.1390 | 0.1667 |
| Ti [wt%] | 0.010 | 0.010 | 0.011 | 0.0103 | 0.0321 | 0.0321 |
| Zr [wt%] | 0.0016 | 0.0016 | 0.0016 | 0.0016 | 0.009 | 0.0096 |

Results

In evaluating the results, it should be noted that the true value of the metal content in the dross is not known.

Sampling technique

Figure 6 shows the cross section of the dross sample taken by cup after saw cut. It consists of mostly metal, even though dross exists on the top layer.



Figure 6 Al dross sample taken with cup from the furnace for charge P2.

The results from the holding furnace are given in 7. The samples with medium and large holes give smaller metal recover than that with small holes. The dross samples from dross pan give similar results as the sample with large holes.

The results from the melting furnace and the holding furnace at the remelter gave similar metal content. Two charges in the melting furnace gave both 41 wt% aluminium recovery whereas skimmed dross from the holding furnace gave 45 wt%.





The aluminium recovery from the dross taken with large holes at the primary producer is shown in Figure 8. Note that we did not manage to take dross samples by medium or small hole sives, since the dross did not drain enough metal with these two. From Figure 8 it is seen that the repeatability of the dross sampling was good, with maximum 12% deviation for various dross samples in the same furnace/dross pan (some with different positions) and maximum 4% deviation with the same dross sample.

For the same charge, the dross samples from the dross pan have lower aluminium recovery compared with the samples taken from the furnace with a large-hole -sieve. For example, it is 38% lower for P1, 20% for P2, 13% for P3, and 10% for P4, where the deviation is not considered.



Figure 8 Al recovery from tests at the primary plant *P for Primary, DP for dross pan, and LH for large hole,*

Primary and remelter results, with and without salt

Figure 9 shows the aluminium recovery from the samples taken from primary producer and remelter for large and medium holes (named LH and MH) from the furnace. In average the aluminium content of the samples from primary is $82\pm6\%$, while remelt samples analyses $38\pm5\%$ with medium and large holes. 44% lower metal content is concluded for remelt from the furnace.





Figure 10 compares the metal recovery from the dross pan from primary producer and remelter. The results from the primary producer gives $67\pm10\%$ metal content, while for the remelt producer $43\pm2\%$, when dross from both the melting furnace and the holding furnace is included. 24% lower metal content is concluded for remelt producer from the dross pan.



Figure 10 Al recovery for dross samples taken from the dross pan. *MF for melting furnace and HF for holding furnace.*

In Table III the aluminium lost to dross is calculated. The value is calculated using the % metal content of dross taken from inside the furnace, and not from the dross pan. It varies from 340 kg to 440 kg per charge.

Table IIII Metal in the dross at the primary plant based on metal content from LH. All numbers are given in metric tonnes.

| Test nr. | P1 | P2 | P3 | P4 |
|-----------------------|------|------|------|------|
| Dross skimmed [t] | 0.40 | 0.52 | 0.38 | 0.57 |
| Al content, LH [%] | 86 | 81 | 90 | 77 |
| Aluminum [t] | 0.34 | 0.42 | 0.34 | 0.44 |

Discussion

Sample remelting

The method of remelting the dross with salt at 1000°C seemed to give consistent results for split samples. That means remelting about 1 kg of dross in the lab scale test can be a reliable, easy and cost effective method to determine the metal content in the dross.

Sampling technique

As shown from the remelter result, the medium-and large-holesieves give lower aluminium recovery than the sieve with small holes. This is the result of better metal drainage through the medium and large holes than through the small holes.

The sampling technique using a large- hole sieve gave surprisingly consistent results for test taken at both the primary production and the remelter plant.

When skimmed the dross into the dross pan, the metal trapped inside the dross would drain. Underestimation of the metal content from the dross pan dross is in addition due to the settling and oxidisation of the aluminium. Overestimation from the furnace dross is due to the trapped metal. That means that although the actual value of the metal content in the dross is not known, one might have an idea about the metal content margins.

Comparing results

The use of salts is the most likely explanation of why the metal content of dross from the remelter is consistent lower than primary producer. This also explains why the remelter dross is more dry than the dross produced by the primary facility. It was impossible to drain the non-salt using dross with medium- hole sieve, duo to the strong interface tension between the dross and melt.

Taking dross samples with the sieve inside the furnace and analysing the metal content could maybe help to link parameters effecting metal losses, such as total energy, furnace atmosphere, cycle time, and alloying elements. It could be used to investigate batch variances on the dross formation in a furnace. For instance the charge P4, where Mg is added just before skimming produces the highest amount of dross and aluminium metal lost in the dross (Table III).

Conclusions

An approximate, but practical and useful method for dross sampling is introduced. The best method for sampling in this study is found to be a sieve with holes that allow metal to drain. Repeatability of the sampling was good. Consistent values for metal content in the furnace dross were obtained when the dross was melted with equal mass of salt at 1000°C.

Apparent difference of metal content in the dross in primary and secondary aluminium production is observed with and without salt treatment. With salt treatment in the furnace, the metal content in the dross is lower.

Measurement of metal content in the dross is important to evaluate metal losses and factors that determine losses. The sampling method allows metal losses to be calculated. In the examples the effect of salt addition on reducing losses is clearly demonstrated.

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