



Review of Dissolution Testing and Alloying Methods in the Casthouse

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International Conference

Continuous Casting

13-15 November 2000 in Frankfurt, Germany

www.metallurgical.com

INTRODUCTION

The properties of Al alloys are largely dependent on the correct addition of the alloying elements before the casting process. There are a number of ways of performing these alloying additions such as pure metal, master alloys, powder injection and a variety of compacted powders additives (tablets, mini tablets and briquettes).

The choice of which alloying addition to use for each element is complex [Thistlethwaite 1992]. A number of competing factors have to be taken into account and their relative importance may vary from plant to plant and product to product. Some of the key criteria include metal temperature available, virgin:scrap ratio, furnace type and layout, addition and stirring practices, alloy change frequency and end product quality. The cost of alloying is not always easily defined. There are not only raw materials costs, but also processing, yield, quality and overhead considerations, which need to be taken into account when selecting the most appropriate alloying technique.

LSM and Bostlan's experience as worldwide suppliers of different products for the aluminium alloying industry is that in recent years consumption of compacted additives has noticeably increased. Some casthouses have stopped their injection production lines, and new facilities for this addition practice are rarely set up, mainly due to capital costs and further the strict quality control requirements of the raw materials because of safety risks when handling powders. Compared to master alloys

additives, compacted powder additives are easy to handle; cold metal quantity to be added to the furnace is not very high (since the lowest concentration of the alloying metal in the compact is 75%); accurate additions for compositional adjustments can be performed if necessary; and stocking costs are reduced.

In the mid 90s some studies on dissolution of compacted powders were published [Young 1993; Campbell 1994; Fisher, 1994; Perry 1994; Shafyei 1995]. Many of these works are focused on laboratory studies, so it can be said that the general mechanism and the behaviour of the compacted additives in small furnaces is known (exothermic heating of the compact, intermetallic compounds and swelling of the compact). During subsequent years, the literature concerning dissolution and recovery of alloying metals from compacted powders has significantly decreased. The most recent work covers deeper studies on the intermetallic compounds influence for the explanations of the dissolution mechanisms [Bristow 1999; Lee 2000].

All this background literature is useful for understanding the behaviour of the compacted additives, but it could be said that in general no recommendations for an industrial practice have been given. There are several issues the casthouse is interested in including dissolution rates and final recoveries in industrial scale furnaces; production of skims/drosses and correct skimming practices for each additive; correct stirring. The challenge is to know how laboratory tests relate to industrial practice.

PRODUCTS

Pressing mixtures of metallic powders (Mn, Fe, Ti, Cu, Cr, Ni, Pb, etc) with aluminium, a flux, or a mixture of both components produces compacted powder additives for the aluminium alloying industry. Alloying metal contents range from 75% to 85%. The most common compacted additives are tablets and mini tablets. Both are cylindrical. Tablets are nominally 90 mm diameter; height and weight depend on the alloying element, but usual figures range from 1250 to 1333 g and approximately 45 mm height. For mini tablets,

40 to 45 mm diameters are available, with 50 to 200 g weights and different heights as well.

Tablets and mini tablets are produced in hydraulic presses, using special steel punches and dies. To maintain acceptable tool life requires the use of a lubricant in the formulation of the product. A comparable product is a briquette with the pressure produced between two compacting rolls with “pillow shaped” indentations in the rolls to form the tablet shape.

TECHNIQUES

The performance and behaviour of compacted additives in molten aluminium furnaces is usually studied by the TP-2 test as published by The Aluminum Association [Aluminum Association 1990], although many laboratories adapt the procedures or equipment to their own facilities. The TP2 test includes the use of crucible furnaces, temperature ranges around $732\pm 10^{\circ}\text{C}$, sampling every minute during the test's first ten minutes, and frequent stirring practices. Following the test, dissolution rates and final recoveries of the alloying metal can be studied, as well as skims produced and reactivity phenomena (bubbles, flames, fumes). Scientific literature [Perry 1994; Shafyei, 1995] mentions also microscopic techniques for the study of the intermetallic compounds, whose formation is the first step before the dissolution.

The connection of these study techniques, and their corresponding application, to industrial practice can be sometimes confusing or not clearly seen, mainly due to problems arising from the different scale when working in the casthouse. New techniques or working methodologies applied to the study of the compacted additives can help to understand the dissolution process.

The microscopic behaviour (the monitoring of what happens in the furnace)

Classical monitoring

Using a steel cone as in the Aluminum Association TP-1 grain refiner test 10 kg of Al is heated in a small resistance furnace. A whole or part tablet is placed on its edge in the bottom

of the steel cone mould. The tablet and mould are preheated and then lowered into the bath and allowed to fill to the level of the notch. The mould is held in the bath for the time required before removing and quenching. The experiment is repeated with different hold times if necessary.

The cast cones are sectioned vertically, bisecting the circular face of the mini tablet. The cut faces of the samples are ground flat to reveal the structure and any undissolved remnants of the tablet.



Figure 1.- Steel cone mould test for 200 g 85%Mn mini tablets. Mini tablets were extracted after 60 seconds, 120 seconds, and 240 seconds after addition

Figure 1 shows some scanned images of the cast samples for 85% Mn-containing 200 grams mini tablets.

Visual information given by this technique is direct. The methodology has not been widely mentioned in the literature [Bristow 1999], but comparisons between different materials can be

performed easily and at relatively low cost by this technique, especially if dissolution rates in the first few minutes are important. Thus, fast checking of the behaviour of the material is possible. As disadvantages, no micrographic studies are possible, and scaling problems could arise when applying results to an industrial furnace. As an example, there is no possibility of using complete standard tablets [only a portion].

X-Ray monitoring

Recently, the Department of Materials of the University of Birmingham has developed a technique that allows a direct view through the dissolution process based on X-Ray radiations. Using this technique, a continuous monitoring of the compacted additive can be recorded on videotape. For the test a mini tablet can be added into a sand mould 125 mm wide and 250 mm deep containing 7 kg aluminium.

This technique confirms directly the results obtained from others, especially those concerning the swelling and breaking down of the compacted additive. Flames in the first seconds are due to the presence of solid lubricants in the formulation of the additives for proper compaction. Further swelling phenomena are due to the formation of intermetallic compounds between the alloying element powder and the aluminium. The additive is finally broken due to the melting of the aluminium/flux within the mini tablet.

This stage does not mean that the alloying metal (for example Mn) has been recovered at this time, but that the compacted structure has been broken down.

The X-ray technique is costly, although direct and comparative results can be quickly obtained. However the dimensions of the sand mould are not adequate for standard tablets or for adding more than one mini tablet as the alloying level would be too high, and swelling phenomena could be uncontrolled. In addition temperature control is not possible during the experiments.

Swollen compacted additives

The techniques described are only useful for direct monitoring of one mini tablet due to the size limitations. Real additions are never like this: many compacted additives are added together usually in the same part of the furnace, so liquid aluminium may not enter into the mini tablet so easily. Swelling phenomena and further metal recovery may thus be delayed.

Swollen compacted additives (one mini tablet, one standard tablet, or some mini tablets) can be obtained from industrial scale test furnaces with a more realistic approach to the customer's situation. As an example, Figure 2 shows some results for 75% Mn 100 grams mini tablets added at 730°C and extracted from the furnace at the times shown. The furnace used was a rotary oxycombustion 400 kg

facility and a 15 cm diameter by 8 cm height holed ladle was used for sinking and extracting the samples. Table 1 below summarises data from the tablet samples.

Time (s)	Temp (°C)	W (g)	h (mm)	Ø (mm)	ρ(gcm ⁻³)
0	N/A	100	17.55	40.05	4.6
30	728-725	103	24.95	49.90	2.4
45	730-728	137	25.00	55.10	2.3
60	731-729	59	N/A	N/A	N/A
70	732-730	23	N/A	N/A	N/A

The extracted and swollen samples can be cut and polished for examining on an optical microscope. Figure 3 shows an example for the piece extracted at 45 seconds. The intermetallic compounds can be seen.

Advantages of this technique include a direct monitoring of the real process in a furnace. Samples extracted can be weighed, and directly analysed. For example, it can be seen that at 30 seconds the swelling phenomena has started but no aluminium has entered into the mini tablet yet. If related to previous techniques (X-Ray), it can be concluded that even if the mini tablet is destroyed at 70 seconds, dissolution is not complete. Conclusions from the micrographic analysis can also be obtained; in this case the samples are closer to what would be obtained from an industrial furnace. Finally, this 400 kg furnace allows also the addition and retention of swollen standard tablets or groups of mini tablets. The main disadvantage of this technique is the cost.

The macroscopic behaviour

In terms of the use of compacted additives, issues of importance in the casthouse include high recovery in a short time, the production of skims and/or dross due to the operation, and the reactivity (flames, fumes, bubbles) of the material added to the furnace. Knowledge of the dissolution mechanism given by the microscopic techniques helps the manufacturer to produce materials with different characteristics for achieving better results. However these results have to be accomplished in an aluminium furnace.

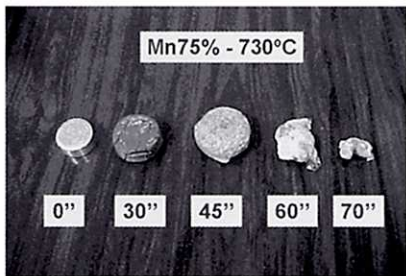


Figure 2.- Swollen samples extracted from the 400 kg furnace

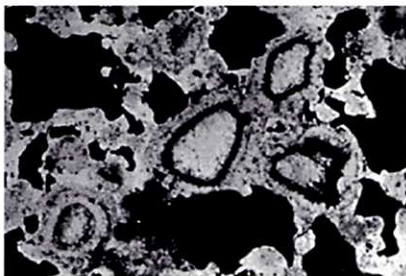


Figure 3.- Microscopy of the sample extracted at 45 seconds (x50)

The Aluminum Association's TP-2 test does not specify any furnace size for the dissolution and

recovery test. Crucible furnaces of any size could be used. Laboratory costs imply that small (10 to 40 kg) furnaces are generally used. The results of these tests are subject to doubt because they are on a much smaller scale:

- Standard tablets cannot be directly studied, since they add too much material for the final alloying level.
- Stirring practices are different to those used in the casthouse. The TP-2 test proposes a highly effective stirring system for every minute of the test for the first 10 minutes.
- Dross production and reactivity have to be estimated in a very small surface. Skimming practices are thus also different from those used in the casthouse.

Issues of relevance to practical dissolution of compacted additive powders cannot be explained just by experiments carried out in small crucible furnaces. With the size of the experiment being such an important factor, a methodology was developed for a 400 kg furnace in order to perform continuous and reproducible dissolution tests. The furnace is shown in Figure 4.

The main aim of using an almost industrial furnace is to obtain results with no scaling problems. The content of this kind of furnace should be stirred for research work, since homogenisation of the melting bath is necessary

for maintaining the temperature and for adequate and repetitive sampling. Stirring is performed before every sampling process, using a rake not hitting the tablets (or mini tablets) added. Samples are usually taken every five minutes for 40 minutes (for Mn, Fe or Cr tests, for example) or even for 90 minutes (for Ti). For performing a proper comparison of the results, materials are added without any packaging, whereas industrial practice is usually to add plastic or foil wrapping, and/or cardboard boxes or paper sacks. Temperature control is performed with a thermocouple sunk in the bath; it is usually accepted to have a range of $\pm 10^{\circ}\text{C}$ for each experiment, but usually a $\pm 4^{\circ}\text{C}$ after material addition can be achieved.

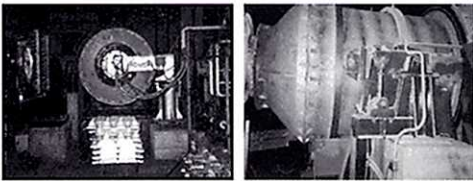


Figure 4.- A rotary 400 kg furnace for aluminium dissolution tests

Some advantages of this facility for reproducing the industrial practices are:

- Realistic stirring practices. A ceramic rake is used in this case, which is longitudinally used for homogenisation. This can be taken

as a very similar practice to that of many customers.

- Distribution studies can be performed. Since differences arise from adding all the material in the same point or in evenly distributed points, a furnace with an adequate surface as this can be used for this kind of research.
- Realistic skimming practices. Experiments can be also performed following the customer's practices for skimming: either before or after addition, or with addition of dressing fluxes if required.
- Furnace capacity ensures the researcher or the customer that any final alloying level of any aluminium series alloy required can be achieved. Addition of standard complete tablets is not a problem.

The main issue with this kind of experiments is the cost, which is affected by the large aluminium quantity used, even if aluminium can be recovered after the experiment. This technique usually yields dissolution curves closer to real situations in the casthouse, but the control of the experimental factors is more difficult and costly than with a smaller (and more easily controlled) furnace.

CONCLUSIONS

The increase in consumption of compacted powders for alloying aluminium in the

casthouse has focused recent research developments in this field. This work has

presented different working methodologies and new-in-the-field applications for ascertaining and proving the behaviour of tablets and mini tablets in aluminium furnaces. It could be said that a supplier controlling these diverse techniques can give a more complete answer to many problems of the customer/producer. In this sense, the most evident application in order to satisfy/answer a customer's dissolution problem is the large capacity furnace. This furnace combines an almost industrial facility with the possibility of performing designed and highly controlled work: working conditions are closer to those of the casthouse, and macroscopic research work can be performed to obtain results concerning dissolution rates, final recoveries, gross production, and reactivity. Dissolution rate results given by this furnace are usually lower than those given by the typically used laboratory scale crucible furnaces. In most cases this should not be taken

as an ineffective method or product, but as a different approach to the problem.

On the other hand, the microscopic monitoring of the behaviour of the compacted additives has been improved with new technologies and different methodologies. Fast and low cost comparative analysis of the dissolution rate can be performed sinking a steel cone mould containing a mini tablet or a tablet portion into a prepared crucible furnace. A direct insight of the swelling and breaking down of a mini tablet can be performed using a sand mould and an xray technique. Fast and comparative (but costly) direct results can be obtained. Finally, different types of real swollen samples obtained from a furnace without scaling problems can be classically studied with the microscope. All of these research results are useful in order to propose new developments/products by the supplier, and to understand specific customer related problems under many different situations.

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