# MELT TREATMENT – EVOLUTION AND PERSPECTIVES

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## Abstract

The use of aluminium alloys is increasingly oriented towards high quality end products, where aluminium enters into competition with other materials on a property/quality/cost basis. The aluminium industry has developed several technologies to provide the required quality of the aluminium alloys as function of the end application. Furnace treatment, in line treatment, and filtration are mainly used to tailor the quality. This paper summarises the evolution of these technologies in the last years, and illustrates the link with quality and product properties. The following aspects are integrated in the discussion: environment, productivity, recycling, flexibility, quality measurement. Finally some areas where developments are required or would be beneficial are discussed.

## Introduction

Melt treatment has been developed as a necessary step for the production of quality aluminium alloys. This is particularly true for demanding applications like can stock, aerospace and high reflectance products which have developed in the last 30 years. Several technologies are available which mainly involve either a gas purging treatment or a filtration of the molten alloy. Selecting the technologies is done based on a combination of the following criteria: quality – productivity – investment and operational cost. The first reason to implement a technology is for improving the quality. In this paper, quality requirements and the development of characterisation technologies are discussed first. Further, the progress in the understanding of the physical processes is illustrated, and the technological developments associated are put into perspective. Finally, some specific trends for future developments are enlightened.

## Quality Measurement

The casthouse products have very different molten metal quality requirement depending on the downstream process or their applications. Hydrogen, alkali elements and inclusion content are the main quality criteria. The selection of the melt treatment technologies is based primarily on expected product quality performance. This requires:
-   The measurement of the quality, preferably on line in the casthouse, and
-   The definition of criteria that link the measured quality to the final property.

A detailed review of available techniques for the assessment of aluminium quality has been done recently [1]. The following comments are oriented towards a user perspective.

With regard to inclusions, the sampling methods (Lais, PoDFA) have been the only options for a long time. They are requiring off line analysis. These techniques allow the inclusions to be analysed, however the frequency of sampling determines the representativity and is most often limited. In line techniques have been aimed at for a long time. LiMCA [2] was the first technology capable of providing a real time on line analysis of both the inclusion content and their size distribution with a good accuracy [3]. The provision of LiMCA resulted in major improvements of the casthouse process knowledge. Furnace and casting related processes were optimised thanks to this technique. Recently, an automatic LiMCA has been made available (figure 1).

However there is still a need for development in the inclusion characterisation domain in order to overcome the limits of existing techniques. The main challenges relate to (a) the measurement after an in-line degasser, and to (b) an increased representativity through higher volume analysis.

(a) In line measurement in presence of micro bubbles is still an open issue. Micro bubbles exit in-line degassers, and LiMCA counting is affected by these bubbles. Although some tricks can be used to guess the bubble frequency [5] or reduce their incidence on LiMCA counting [6], there is no reliable solution to make LiMCA in the presence of micro bubbles. Several probe shapes are provided, but none is unaffected by micro bubbles, and the readings depend on the shape of the tube [6].

(b) Current available techniques analyse a limited amount of the metal cast. For LiMCA it is typically 0.01 vol % of the metal cast that is being sampled. Ultrasonic detection has been seen as a potential improvement as it allows the analysis of a much larger volume of metal during a cast [7]. Although progress is made, questions are still open with regard to both the absolute number and the size distribution of inclusions provided by the ultrasonic analyser.



Figure 1: LiMCA CM [4]

With regard to the hydrogen content of the melt, Alscan is the reference technology [8]. Based on an accumulation loop it provides a data typically every 6 to 15 minutes. Very reliable operation can be obtained once a standard analysis procedure is defined [9]. Recent analysis studied the influence of the air atmosphere on the equipment and suggested part of the humidity dependence of hydrogen in the melt may be linked to the equipment itself and not to real variations of the hydrogen content in the melt [10]. This aspect is currently under further evaluation. Having a fast response probe would allow understanding fast kinetic or transient phenomena (start phase of a cast,…). Developments are currently done on electrochemical probes [1].

With regard to the main measurement techniques used today (LiMCA and Alscan), it should be reminded that they provide a quality index, which is not an absolute value. Standard measurement procedures are not defined, and it is known that several factors related to the measurement procedure affect the result. For Alscan measurements, the acquisition duration, the number of data used for a hydrogen measurement, and the time into the cast have an influence [9]. For LiMCA measurements, the

kind of measurement tube and the presence of micro-bubbles have been illustrated as major factors [5, 6], the procedure used for the analysis of the data also has an influence. For these reasons, comparing absolute figures must be done with care as the conditions of the measurement are not often reported.

Moreover, these equipments do not allow the characterisation of the product quality, as they analyse a fraction of the metal. The characterisation tools are most often used for process validation, or R&D. The need for characterisation equipments allowing the product to be fully controlled in-line is still present. Finally, the establishment of the correlation between a casthouse quality index and a final property depends on the customer's need and cannot be generalised.

### Gas Purging – Physical Basis

The physical basis of the gas purging have been known for a long time [11]. In these treatments, a carrier gas is used (argon, sometimes nitrogen), and a reactive gas is often added (mostly chlorine, others have been evaluated). It is only recently that the measurement of the physical parameters affecting the process has been initiated in aluminium, given the discrepancies observed with previously used water model tests. The focus was set on the understanding of the process performance and of the phenomenon that affect the gas purging process.

### a. Gas Bubbles

The distribution of gas bubbles in a reaction volume is the basis of the operation of a degasser. Although global parameters could be measured (like gas hold up…), it is only recently that physical data have been collected on the bubbles directly inside the treatment zone.

The bubble size is a major parameter, as it will determine both the surface offered to the reaction and influence the residence time of the bubble in the reactor. Recently, the use of X-Rays to view bubbles inside molten aluminium has allowed the determination of parameters like bubble size and bubble rise velocity [12]. It has been shown that the bubbles were in the range of 6 to 40 mm, depending on the wetting properties of the substrate, when the diffuser was a static tube (figure 2). The rise velocity was measured to be in the range of 0.35 – 0.45 m/sec, with only a slight influence of the bubble size.
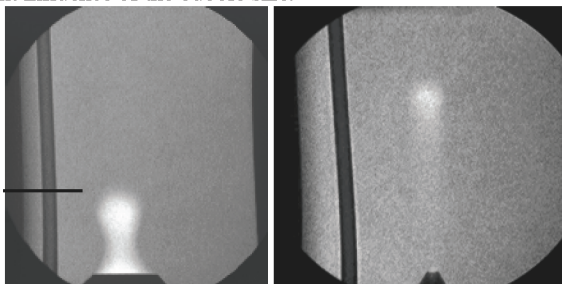


Figure 2: Bubble generation on a static diffuser. Non wetting diffuser (left) and wetting diffuser (right) [12]

Measuring inside a stirred tank is even more difficult. A capacitance probe has been developed [13]. The probe has provided an insight to the distribution of the bubbles in the degasser. It was shown that the bubble frequency decreased when the distance with the rotor was increased. The measurement of bubble size and also of a bubble size distribution, was still shown to be difficult, recently an acoustic detector has been suggested

therefore [14]. Other aspects under study relate to the surface of the degasser. Work on the formation of droplets ejected at the metal surface when a gas bubble explodes has recently been initiated with a model alloy system [15].

### b. Degassing - Equilibrium Hydrogen Content

The Sievert's law predicts the equilibrium hydrogen content is a function of three parameters: the metal temperature, the alloy composition and the humidity content of the surrounding air. Transferred into the Alscan, the equation expresses as:

$$H_{eq} \equiv C_{\underline{H}} = 0.92 \bullet C_A \bullet \exp\left[6.531 \bullet \frac{(T - 700)}{(T + 273)}\right] \bullet \sqrt{AH}$$

$$C_A = 10^{(0.0170 \bullet wt\%Mg - 0.0269 \bullet wt\%Cu - 0.0119 \bullet wt\%Si)}$$

The ambient air humidity is the major parameter of practical importance. Several publications have illustrated the influence of humidity content in air on the hydrogen after a degasser among which [9, 16, 17] (figure 3). Although the extent of the relationship may be questioned [10], the influence of this parameter is also established based on the final properties of the products. There are still discrepancies in the literature as to how the ambient humidity has to be expressed. As long as condensation is not involved, the content of humidity in air is expressed taking into account both the relative humidity of air and the air temperature.
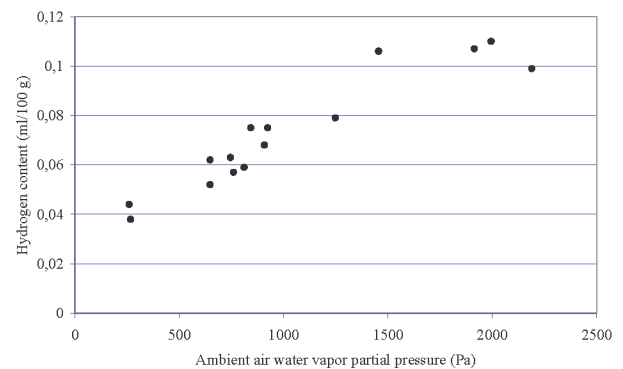


Figure 3: Relationship between the hydrogen content after the degasser and the ambient air humidity [9]

### c. Inclusion - Flotation

Inclusions present in molten aluminium differ widely: shape, size, density, nature. In gas purging processes, it is considered that the major inclusion removal mechanism is flotation. However only limited data are available with regard to the detailed mechanisms that occur during the process. Chlorine is recognised as a major element for an efficient inclusion removal, as neutral gas alone cannot remove inclusions efficiently [18]. Chlorine reacts with Mg, when present in the melt, and magnesium chloride forms. These chlorides have been reported to promote agglomeration of inclusions, which is demonstrated in particular with refining agents [19]. Kinetic factors affecting the removal of inclusions by flotation have been studied [20]. The influence of the chlorine concentration, gas amount and stirring energy were analysed.

Quantifying inclusion removal by a degasser is uneasy. The sampling method most often used is PoDFA. LiMCA results have

been reported in detail in [5]. Efficiencies were indicated to be in the range of 50% for 20 μm inclusions, 98% for 40 μm inclusions. These data are reasonable estimates given the probable contamination of LiMCA counts by micro-bubbles.

Inclusion flotation in a degasser unit is an area in which more understanding could be gained, in particular with regard to the influence of the free surface and dross build up in the reaction zones.

### d. Alkali and Alkaline-Earth Elements Removal

The removal of alkali and alkaline-earth elements is efficiently performed by gas fluxing, the halide elements (chlorine and fluorine) being prone to react with Na, Ca and Li. This process is well known and abundant literature is available on this topic [18]. Comparisons between different technologies show that the rotary fluxing technology is the most efficient [21]. Recent developments have targeted the use of salts instead of gaseous chlorine. Rotary flux injection in furnaces has been used for years, with performances very similar to those of chlorine injection [22]. It has been shown recently that an in-line salt based process is able to reduce Na and Ca to the desired levels, although the kinetics is slightly slower than with gaseous chlorine [23].

### e. Modelling of the Gas Fluxing Processes

A few years ago, models were able to describe the macroscopic phenomena, and the results could be compared with macroscopic measurements (for instance, degassing efficiency, inclusion removal rate...) [24]. The progress of the calculation power and the development of the calculation codes have allowed a significant progress of the modelling of the gas fluxing processes. Furnace fluxing has been modelled with local hydrodynamic description, and validated through the global efficiency of alkaline earth element removal [25, 26]. Recently developed models have been validated with experimental measurements of bubble distributions in the reactor [27], bubble sizes and shapes and local mass transfer coefficients [28].

Future developments will probably be able to better describe both the local hydrodynamic behaviour and the local physical processes. This will be particularly helpful to take into account the interfacial reactions: free surface of the melt, surface of the gas bubbles, rotary impellers, walls of the containers. However these progresses will be helpful only if an experimental database is built which will provide the needed data to assess the validity of the modelling progress.

### Furnace treatment - Technology development

In the past, lances have been used as a major treatment technology (figure 4). Nitrogen or argon is introduced in the melt together with chlorine which is considered needed for metal cleaning. Very high proportions of chlorine in the treatment gas were common, and sometimes pure chlorine was used. Given the low efficiency of lances, there was a desire to obtain a higher mixing of the treatment gas with the aluminium melt. This is achieved through rotary gas injection, through the roof [29] (figure 5) or through a side wall [22, 30] (figure 6) of the furnace. Porous plugs have been used for long in casthouses, their reliability has been increased over time [31] (figure 7). The developments towards a better distribution of the gas in the melt have allowed both a reduction of the treatment durations and a reduction of the chlorine content in the melt. Today, furnace treatments of 30 min,

with a gas mixture containing less than 20 vol % chlorine are common. The trend in the last years has been towards the cancellation of gaseous chlorine, due to environment, health and safety constraints.


Figure 4: Lance treatment in a casting furnace


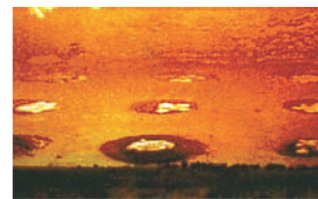Figure 5: IRMA [29]


Figure 6: RFI technology


Figure 7: Porous plugs

### In-Line Degassing - Technology Development

The in-line degassing process has been available for almost 40 years in aluminium casthouses. The development of high quality end uses for aluminium, including can stock and new aerospace alloys, has oriented the development of the technologies towards increased degassing and inclusion removal performances. This has been achieved through internal design of the treatment chambers, and in particular through the design of the gas dispersion equipment. New rotor designs were aimed at an increased shearing action combined with a reduced surface turbulence. High flow-rates are accommodated trough multi-staging.

In the 90', the developments have been oriented towards cost reduction together with an increased operational flexibility. Until then, most degassers had a dead capacity of typically 1 – 3 T, and required a draining operation or a flush for most important alloy changes. Emphasis has been put on the ability to change alloy, with either self draining trough degassers, or tiltable degassers. In terms of contact time, two orientations where followed:
- Low treatment volume, with a higher free surface/volume ratio (figures 8 and 9).
- High treatment volume, with a reduced free surface/volume ratio and a long contact time (figures 10, 11 and 12).

For new equipments it is highly desirable that they are drainable and that the metal drained may be considered as high quality.
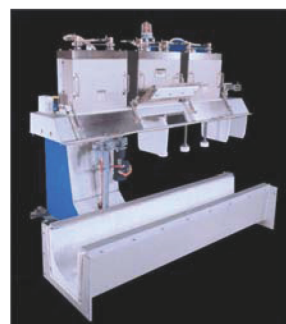

Figure 8: Self draining ACD – Low contact time


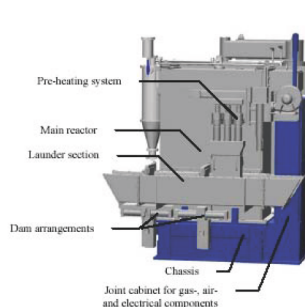Figure 9: Self draining Jet Stirrer – Low contact time [32]

Figure 10: Self draining SIR –
High contact time [33]



Figure 11: Draining Alpur
with rotating spout – High
contact time



Figure 12: Draining LARS with flexible spout – High contact
time [34]

## Filtration – Physical basis

The industry relies on ceramic foam filters (CFF) and deep bed
filters (DBF) to obtain the quality required for their end products.
The efficiency of CFF has been shown to vary widely, the filter
grade being a major factor [35]. Experiments to understand the
inclusion capture in CFF showed that inclusions concentrate in the
entry side of the filter (figure 13). Bridges form in recessed
passages, favouring inclusion agglomeration (figure 14) [35, 36].
It has been shown in 1050 alloy casts that the addition of the
refiner upstream of the CFF lead to significant inclusion releases
from high grade CFF at high inclusion loads [37]. The same effect
has been reported on high Mg alloys [35].
DBF have the potential to provide very high efficiency. It has
been shown that the performance of the filters slightly reduces
with the number of charges cast with a bed [38, 39]. The
deposition of inclusions on a water model of a deep bed filters has
been studied [40]. Conditions for releases were observed, the start
and stop periods appearing critical.
Like for gas purging, modelling has developed in the last years to
describe the physics inside the filters. For CFF, X-Ray
tomography of the filter provides the structure to model (figure
15), and local interactions are described [41]. DBF models
describe the efficiency and the releases [42].
The releases that occur subsequently to hydrodynamic pressures
or for any other reasons limit the reliability of the filters. Reactive
filters have been considered in order to enhance inclusion
adhesion on CFF, however limited data is available on their
applicability and performance [43]. There is still a need to further
develop the knowledge of inclusions life inside the filter in order
to increase the reliability of the filters and to provide the physics
needed to increase the validity of the models.

## Filtration - Technology Development

Both CFF and DBF filtration technologies are well established for
years. For CFF, filters ranging from 30 to 80 ppi are used in

commercial operation. Based on the incidence of the grain refiner
on the CFF efficiency, the XC filter has been developed which
incorporates both a reusable CFF and a DBF, with refiner added
before the DBF [36]. Such filters are believed to improve the
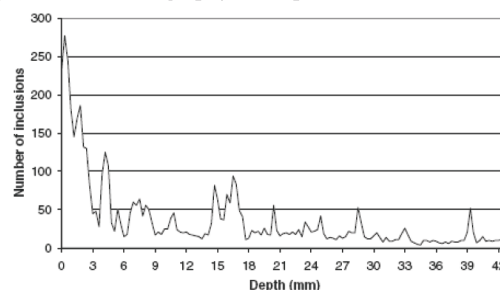quality of foil and lithography sheet products.



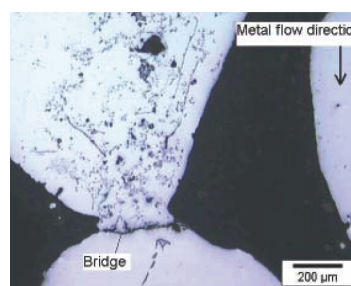Figure 13: Inclusion repartition in the depth of a CFF [35]



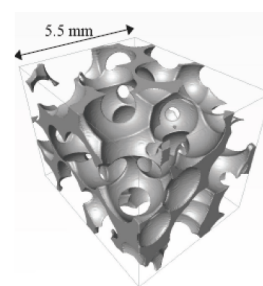Figure 14: Bridge forming in a 50
ppi CFF [35]



Figure 15: Reconstructed
3-D structure of a CFF
filter [41]

### Future Trends in Metal Treatment

Today existing technologies allow the producer to obtain the
required performances. Some technological progresses are
currently been done which will likely affect some of the metal
treatment steps. The following illustrates a few of these possible
evolutions.

a. Gaseous Chlorine Suppression

Chlorine has been extensively used in casthouses as a cleaning
agent, improving the inclusion cleanliness [18]. The reaction
mechanism involves the formation of chloride particles by
reaction with alkali or alkaline-earth elements. These salt particles
increase the flotation performance of the process.
Chlorine by itself is a poisonous agent, and chlorine emissions
may be a concern. The factors influencing the generation of
chlorine/chloride containing emissions have been studied [21, 44],
and the compliance with regulations is a priority [45]. Although
some processes have been run extensively without any chlorine
agent, most of the casthouses were using chlorine at some stage of
their process. The reduction of chlorine consumption, and if
possible the suppression of chlorine from casthouses, is a
desirable objective for environment, health and safety reasons
[16]. The first steps were the cancellation of pure chlorine
treatment and replacement by diluted treatments, followed by
progressive reductions of the chlorine proportions in the fluxing
gas. Significant reductions of the yearly consumption were
obtained doing so; examples of reductions by a factor of 5 to 10
have been achieved [29]. Strategies for the reduction of chlorine
consumption have been proposed [21]. The direct incorporation of

chloride salts carried by a N2 or Ar flow has been considered for the replacement of chlorine in furnace treatment. Casthouses have already banned the use of chlorine for furnace treatments for years, and rely on salt based processes [22]. Recently a process has been demonstrated that allows the replacement of chlorine by salt for an in-line degasser (figure 16).

The possibility to suppress chlorine in the casthouse has been a major achievement of the last years. However the replacement of an existing process by a salt based one is a significant change. It is believed that the suppression of chlorine or the replacement by salt will remain a major focus in the future.
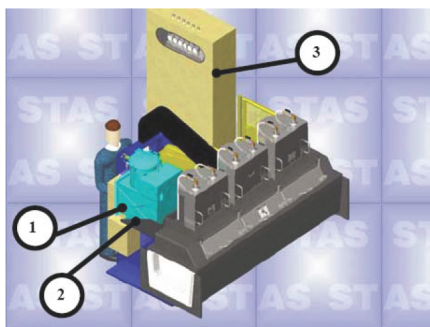


Figure 16: In-line ACD salt fluxing [23]

b. Flexibility Requirement

The casthouses are facing an increasing requirement for high flexibility. Reasons for that are the requirements of reduced inventories, of just in-time delivery to the customers, and the shorter order batches. Casthouses that were used to produce long runs have to rethink their treatment sequences in order to maintain high quality at low cost. Flushing of equipments has become a significant cost contribution. Low metal hold-up equipments are needed insuring a high quality. The flexibility requirement is expected to increase further in the future.

c. In-Line Composition Adjustment

Having the opportunity to measure in-line the composition of a molten metal during the cast technique would induce modifications of the process control strategy, or new processes to be developed. LIBS has been considered for a while, and evaluations in molten metal have been reported [46]. As a first application, the monitoring of the performance of in line treatments would be made possible (Na removal,…). The alloying practice may also be reviewed, for instance for Mg additions. Local additions of specific elements have also been reported and an in-line analysis would allow this process to be monitored.

Such process evolutions will require a reliable cost efficient in-line analysis to be available. The impact on melt treatment strategy would be significant.

d. Purification

Melt purification has been oriented towards the removal of hydrogen, alkali and alkaline-earth elements, and inclusions. Only few metallic elements are removed: Mg, Ti or V. Work has been started in order to evaluate the potential removal of metallic elements like Fe, Mn, Si [47, 48]. Any such purification process will require technologies able to remove a much higher amount of second phase than currently. Calculations show that the particles to be removed will be in the range of volume percent of the

flowing metal, as compared to ppm when conventional inclusions are concerned. Separation processes able to achieve a high recovery have to be developed and integrated in the casthouse organisation to make it a cost effective process.

**Conclusions**

Melt treatment has developed in the last years into a necessary process for any high quality aluminium product, involving furnace fluxing, in-line degassing and filtration. It has been shown that recent evolutions have been oriented towards cost efficient processes, under quality control. The development of quality monitoring, in particular related to inclusions monitoring, would be a push towards the right quality for each product. Future developments are expected and some possible areas have been suggested to meet the sustainable growth of the aluminium industry and the extension of recycling.

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