# NONMETALLIC INCLUSIONS IN THE SECONDARY ALUMINUM INDUSTRY FOR THE PRODUCTION OF AEROSPACE ALLOYS

Bernd Prillhofer<sup>1</sup>, Helmut Antrekowitsch<sup>1,2</sup>, Holm Böttcher<sup>3</sup>, Phil Enright<sup>4</sup>

<sup>1</sup>Christian Doppler Laboratory for secondary metallurgy of non-ferrous metals, Franz-Josef-Str. 18, Leoben, 8700, Austria

<sup>2</sup>Chair for non-ferrous metals, University of Leoben, Franz-Josef-Str. 18, Leoben, 8700, Austria

<sup>3</sup>AMAG rolling GmbH, post office box 32, Ranshofen, 5282, Austria

<sup>4</sup>N-Tec Limited, Watery Lane, Hook Norton, Oxfordshire, OX155NW, England

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## Abstract

Due to enormous growth of the secondary aluminum industry and the required increase in melt quality, the development of methods for inclusion removal has become highly important. To produce high quality aerospace alloys with very low inclusion contents, casthouses must analyze and optimize their production processes from the beginning to the end. Nowadays, there are several methods available for process evaluation and the determination of inclusion level from one step to another. Furthermore, the most important parameters for control of the final inclusion level have to be investigated.

This paper characterizes the change of the inclusion content for the alloy AA 7075 during standard casthouse processing. The possibilities for the improvement of the melt quality are also discussed.

#### Introduction

The casting facility which is going to be discussed is used for the production of 2xxx-, 5xxx- and 7xxx-series rolling ingots. These ingots are rolled to sheets and plates.

Due to the high safety standards for applications in the aerospace industries, plates which are used as a raw material for components have to undergo an ultrasonic check. In order to reach the safety standards, the material has to be free of inclusions above a critical size. Because of this, the main focus during casthouse operations is a reduction of the inclusion content from one step to another. This paper discusses the process of evaluation for improvements to the alloy AA 7075.

For this alloy, the generally large variety of inclusions [1] can be reduced to three main groups:

#### 1. Nonmetallic inclusions:

- Al<sub>2</sub>O<sub>3</sub>, MgO, Al<sub>2</sub>MgO<sub>4</sub> in compact form or as cluster
- Material from refractory lining
- TiB<sub>2</sub>-Agglomerates (exact type depends on the grain refiner used)
- Salt
- Reaction products with graphite components

## 2. Metallic inclusions:

- Cr-, CrMn- and Zr-aluminides
- Incompletely dissolved alloy elements
- 3. Oxide films:
  - Al<sub>2</sub>O<sub>3</sub> films
  - MgO films

All the investigations have been performed in co-operation with Austria Metall AG (AMAG), Austria and in part with N-Tec, England.

#### Process layout

The casting facility at AMAG for the production of 7xxx series ingots consists of a single melting furnace, two casting furnaces, a SNIF P140, a ceramic foam filter unit and an electro magnetic casting pit for 4 ingots (see Figure 1).



Figure 1. Process layout EMC asset group

The raw material which consists of more than 50 percent in-house scrap and of primary aluminum is melted in a gas fired and tiltable melting furnace with a capacity of 33 tons. Subsequently, the melt is transferred to one of the two casting furnaces (33 t capacity) and alloyed. The casting furnaces are channel induction furnaces. After this step, the melt is skimmed and stirred by a gas lance purging treatment. To reduce the amount of alkali metal, dissolved hydrogen and inclusions, a chlorination treatment with a FDU/RDU [2,3] rotary degasser is carried out. Further, the melt is then skimmed again and trimmed with respect to the chemical composition. Finally, after a certain settling time, the casting process starts. To separate large dross particles, a glass cloth channel filter is positioned in front of the SNIF P140 unit. Afterwards, the melt is grain refined and filtered through a ceramic foam filter (CFF) before the melt is distributed to one of the four electromagnetic molds.

#### **Experimental setup**

To evaluate melt purity, two standard methods are used. Both are pressure filtration techniques, Prefil<sup>®</sup> and PoDFA.

# 1. Prefil<sup>®</sup> - a shop floor technique

The Prefil<sup>®</sup> test uses the flow-rate of molten metal through a micro filter at constant temperature and pressure, to measure the quality of the metal. Very clean metal flows quickly, giving a steep straight line in the test output. Inclusions in the metal, such as oxide films, quickly build-up on the filter surface during a test and reduce the flow-rate through the filter.

The Prefil<sup>®</sup> operating principle is shown in Figure 2 with a detailed explanation given in [4-7].

#### 2. PoDFA - a metallographic method

Although it is very time consuming, metallographic examinations for inclusion analysis have to be carried out. PoDFA and Prefil<sup>®</sup> data can be correlated to connect the filtration curves with the total inclusion content (TIC) and to create a database. After several tests, it is possible to estimate the TIC value from the Prefil<sup>®</sup> characteristics alone.

The PoDFA operating principle is explained in detail in [4,7-8].

#### Evaluation of melt cleanliness

To characterize the final melt quality, PoDFA samples were taken at a certain period of time, particularly after the CFF. Due to the fact that the melt quality was found to vary a number of process improvements were attempted and evaluated with the Prefil<sup>®</sup> testing method in combination with metallographic analysis of the filtration residues.

To enable direct comparison between different Prefil<sup>®</sup> curves without doing PoDFA analyses, two Prefil<sup>®</sup> curve parameters for each have been calculated and compared.

The first parameter is the "*Total Area under Prefil*<sup>®</sup> *curve*" which is calculated by finding the total area under the Prefil<sup>®</sup> curve within the standard testing window (150 s and 1400 g) and has the unit of kg·s.

The second parameter is the "*Prefil*<sup>®</sup> curve gradient 3 - 21 s". It has been observed that the initial gradient of a Prefil<sup>®</sup> curve is largely influenced by medium to large inclusions such as oxide films, refractory particles and inclusion clusters, but not by well dispersed grain refiner particles. Such small particles cause the

curve to turn over after approx. 30 s. Therefore, the initial gradient between 3 and 21 seconds can be used to give a quick indication of whether fine particulate is present.

The current process can be divided in several steps inbetween which Prefil<sup>®</sup> tests have been carried out:

- 1. after melting the charged material
- 2. after alloying Mg in the melting furnace
- 3. after melt transfer and skimming
- 4. after lance treatment
- 5. after chlorination treatment with the impeller
- 6. after final adjustment of the chemical composition + lance treatment
- 7. after settling time
- 8. after SNIF and before grain refinement
- 9. after CFF

# Testing conditions

All Prefil<sup>®</sup> tests were carried out at 685 °C and a pressure of 12.1 psi. The PoDFA tests were carried out between 680 and 690 °C and a pressure of 12.1 psi. The filtered melt weight for the PoDFA samples is 1000 g.

### Melt cleanliness - before process improvements

With regard to process improvement, it is very important to know the current position before starting investigations to enhance melt purity. At AMAG, PoDFA samples have been taken nearly every two months to check the melt quality. Sampling positions were always in the launder during the casting process as follows:

- 1. before SNIF
- 2. after SNIF & grain refiner
- 3. after CFF

Figure 3 shows the average decrease in inclusion concentration (based on the average TIC of the melt after finishing melting) for the process steps mentioned above. It can be seen that the loading after melting the charged material is rather high in comparison with the amount after the final refinement steps.

Easy Data H



Figure 2. Prefil<sup>®</sup> operating principle [3]

The in-line treatment does a very good job and decreases the loading to less than 7 % after SNIF + grain refinement and to less than 5.5 % after the CFF.



Figure 3. Melt quality before process improvement

#### **Results and discussion**

# Process improvements

For the final melt quality, it is very important to understand that the inclusion levels, particularly oxide films and metallurgical spinel, should always be minimized at every stage of the process. If operating properly, all metal cleaning processes are percentage processes, so generally, "cleaner in" means "cleaner out". This leads to the conclusion that every step of the production chain has to be optimized. Accordingly several procedures are carried out to improve melt cleanliness step by step.

In addition, there are a number of potentially deleterious operations which must also be carried out. Even through a decrease in melt quality may occur as a result e.g. when transferring the melt from one furnace to another. Therefore, careful handling and decreasing the negative influence is also absolutely necessary.

When trying to reduce the total inclusion content, mechanisms about inclusion development should be known and understood. AA 7075 is an alloy with an average magnesium content of 2.5 %. Due to the relatively high affinity of both – magnesium and aluminum – to oxygen, oxides are going to be produced at every situation where oxygen is present. Hence, all PoDFA samples show a high percentage of magnesium oxide, the reaction product with aluminum oxide – spinel – and oxide films (see Figure 4). Beside this, certain amounts of the total inclusion content are carbides and grain refiner particles. The origin of carbides is primary aluminum, breakdown products from oily scraps as well as products due to reaction with graphite components e.g. impellers.

Based on this, a number of improvements have been developed or considered for incorporations into the standard production process.

### These are as follows:

1. A new concept for alloying magnesium has been developed.

It differs in the addition procedure and considers the burn-off of magnesium.

- 2. Due to the well known effect of dross generation during lance treatment and the potential risk of oxide entrainment, this step has been shortened to a minimum and placed in the right order of the process flow.
- 3. As a result of the poor results out of the Prefil<sup>®</sup> tests after the chlorine conditioning with the impeller, a higher efficiency rotor blade is currently being developed.
- 4. Finally, trials have been carried out to decrease porosity of the ceramic foam filter.



Figure 4. PoDFA sample from the casting furnace, showing dirty metal with fine carbides, magnesium oxide, spinel particles and oxide films

## Melt cleanliness after process improvement

Because PoDFA analyses are very costly and time intensive before a clear result can be reached, Prefil<sup>®</sup> tests have been done to rapidly assess the effect of process changes.

Starting with a baseline process evaluation a certain amount of test residues have been metallographically analyzed. In this way it has been possible to correlate the Prefil<sup>®</sup> curve and especially the two curve parameters mentioned above with the total inclusion content.

A statistically firm amount of drops has been accompanied doing Prefil<sup>®</sup> tests between each step of the process. Afterwards, the total area under the curve, as well as the gradient between 3 and 21 seconds have been calculated.

Figure 5 shows the average result of every operation:



Figure 5. Prefil<sup>®</sup> results of different process steps

process step	area	change in area	gradient	change in gradient
	[%]	[%]	[%]	[%]
after melting the charged material	100,0		100,0	
after alloying Mg in the melting furnace	93,7	-6,3	101,8	1,8
after melt transfer and skimming	90,3	-3,4	93,9	-7,9
after lance treatment	102,3	12,0	102,7	8,9
after chlorination treatment with the impeller	90,8	-11,5	102,4	-0,3
after final adjustment of the chemical				
composition + lance treatment	104,2	13,4	109,7	7,3
after settling time	107,4	3,2	117,4	7,7
after SNIF and before grain refinement	124,1	16,7	132,4	15,0
after CFF (standard)	120,8	-3,4	115,4	
after CFF (standard + 10 ppi)	125,1	1,0	138,2	5,8
after CFF (standard + 20 ppi)	127,8	3,7	135,8	3,5

Table I. Data of average Prefil<sup>®</sup> curve parameters for each process step

The results relate to the initial values after melting the charged material which is set 100 %. So one can see that values below 100 % illustrate a decrease in melt quality. On the other hand enhanced melt cleanliness is given by an increase of both, area and gradient step by step. To demonstrate the efficiency of each element of the production chain, the variation of area and gradient has been calculated. Detailed data is given by Table I.

Metallurgical spinel, magnesium oxide and fine carbides are the major inclusions found in the melting and casting furnaces. The level of these inclusions is generally high, although the final treatment in the casting furnace has a significant cleaning effect. Oxide film levels are also high in the melting and casting furnaces, but are heavily reduced until after "in-line" treatment.

Alloying magnesium in the melting furnace leads to, as expected, an increase of magnesium oxide due to the melting loss. This effect can only be minimized, but not removed completely. Also, an increase of metallurgical spinel can be observed because of the reaction of magnesium oxide with aluminum oxide.

Transferring the melt from the melting into the casting furnace in combination with most of the alloying work, results in melt quality reaching a minimum. This can be attributed to high levels of turbulence and the entrainment of inter-connected oxide films, as well as the input of impurities from the alloying ingredients.

After skimming dross, a lance treatment is applied to homogenize the melt and make sure that all alloying elements are dissolved. Although dross is going to be generated because of the surface turbulence, injected gas bubbles lead to a decrease in inclusion content through the flotation effect.

A further chlorine treatment which is used primary to reduce alkali metal residues, dissolved hydrogen and to do another melt homogenization, has a detrimental effect on the Prefil<sup>®</sup> characteristic. This is attributed to the inefficient geometry of the rotor blade and caused problems in connection with this.

The lance treatment after final correction in chemical composition effects an increase in the melt quality.

There is only a minor effect of settling in the casting furnace. This is not unexpected, due to the channel inductor.

The metal quality after the SNIF is generally good but a carry through of all inclusion types is observed. The throughput of inclusions decreases highly by providing melt with an increased melt quality before the treatment. Although the inclusion content after the CFF is very low and within generally acceptable limits, using standard porosity, the inclusion profile is similar to that observed before the in line treatment. That is metallurgical spinel, magnesium oxide and fine carbide particles are all still present. In this case, a trace of titanium diboride (grain refiner) is also present – as expected following grain refiner rod injection.

The Prefil curves show a significant improvement in the metal quality after filtration with 10 ppi or 20 ppi finer filters compared to standard use.

Comparison before and after process improvment

To draw a comparison before and after implementing arrangements to improve the final melt quality, the total inclusion content after using a standard + 20 ppi ceramic foam filter was decreased to a minimum of  $0.001 \text{ mm}^2/\text{kg}$  (see Figure 6).



Figure 6. PoDFA sample from after the CFF, showing clean metal

In general, to highlight the differences between the old and the current process (see Figure 7), the melt leaves the casting furnace much cleaner than before. A reduced inclusion loading in the melt leads to a better performance of the final melt refinement. SNIF and the CFF are an effective metal cleaning combination. Both total inclusion contents and oxide film levels fall to very low levels after the in line treatments.



Figure 7. Melt quality before and after process improvement

#### **Operating windows**

In casthouse operations, it is very important to know whether metal quality is inside or outside a specification. Depending on this, operators make the decision 'Go/No-Go'. Relaying on a Prefil<sup>®</sup> test to help make this decision requires an "operating window" for every part of the process. Furthermore, all information provided by the test curves has to be well interpreted. Clean, un-grain refined metal results in a Prefil characteristic that is essentially a steep straight line. The presence of small oxide films results in a steep straight going at the beginning and a final turn off. The addition of grain refiners introduces small titanium diboride particles to the melt. The presence of borides causes the Prefil characteristic to deviate from a straight line and produces a rate of change of curvature that is dependent on the boride loading (see Figure 8).



regarding to different loadings [9]

## Melting and casting furnaces

Prefil<sup>®</sup> curves are usually interpreted by comparison with an industrial range or production window. The real benefits of Prefil<sup>®</sup> are realized when the Prefil<sup>®</sup> curve for a particular sample, obtained on-line, is compared with a pre-established window or footprint for appropriate quality.

Operating windows, whether for a process group or a single process step, are used to control the melt quality, treatment efficiency or while doing process improvements. It can be derived by plotting the average Prefil<sup>®</sup> curve, resulting from several tests plus and minus the standard deviation.

In this case, two operating windows have been selected for the process steps in the melting furnace (see Figure 9) and the casting furnace (see Figure 10). These windows are shown below



Figure 9. Operating window melting furnace

Comparing Figure 9 and 10, one can see that after melting the charged material, the various process operations lead to a wider Prefil<sup>®</sup> window indicating less consistency in the metal quality.



Figure 10. Operating window casting furnace

## Launder

Doing a final quality check after the CFF enables the operator to decide whether the product can be used for critical plate production or not. In cases where melt quality is not sufficient enough, the ingot can be used for applications which are less inclusion critical.

From this study, the operating window for the in-line treatment (see Figure 11) is a very powerful tool for decision making. The left border of the operating area represents a total inclusion content of  $0.001 \text{ mm}^2/\text{kg} \pm 40 \%$ .



### Conclusion

Overall, one can see that there is a clear difference between the quality of the metal in the melting and casting furnaces, as well as after the in-line treatment processes (see Figure 12). Yet, this step change also shows that the in-line processes are working well.



Alloying and treatment practices in the casting furnace are an important part of the metal cleaning process, and this is where most of the inconsistency in metal quality is likely to originate.

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