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DOCTORAL SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

PhD THESIS SUMMARY

INDUSTRIAL EXPERIMENTAL RESEARCHES ON THE EVOLUTION OF THE LEVEL OF INCLUSIONS IN ALUMINUM AND ITS ALLOYS DEPENDING ON THE ELABORATION AND CASTING CONDITIONS

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CONTENT

ABSTRACT

The main objective of the doctoral thesis is to improve the quality of aluminum cast products and its alloys, as well as to increase the performance of the technological casting process operations, through the development of high efficiency systems, reducing the level of inclusions in the final product being one of the methods that can lead to the achievement of this goal.

The reduction of the content of inclusions in the metal bath is achieved by using fluxes – solid salts or mixtures of solid salts. With these salts the inclusions are transported to the surface of the metallic bath to be removed. At the same time, in the production flow, in reducing the level of inclusions, a very important role is played by sedimentation and flotation, degassing, refining (by adding a refiner) and filtering the metal. All these methods combined and done properly can lead to a better internal quality of molded products. The main source of rejection of cast aluminum products and subsequent non-compliant processing is the inclusions from the aluminum melt.

Industrial experimental research was carried out with the help and agreement of the collective from Alro S.A. Slatina

Keywords : inclusions, flux, salts, sedimentation, flotation, filtration

PART I
DOCUMENTARY ANALYSIS OF THE CURRENT STAGE OF RESEARCH REGARDING THE TYPES OF INCLUSIONS PRESENT IN ALUMINUM ALLOYS

CHAPTER 1. TYPES OF INCLUSIONS IN ALUMINUM ALLOYS. MECHANISMS OF FORMATION OF INCLUSIONS

1.1. Introduction

Light alloys, which include aluminum alloys, play an important role in today's industrial community. Even with the advent of new nanocomposite materials and innovative ceramics, aluminum takes second place in use, after steel.

Its light weight is its greatest advantage, in addition to the special advantages of aluminum casting alloys, which are relatively low melting temperature, insignificant solubility for all gases except hydrogen, and the good surface finish usually obtained in products finals. [1]

The experimental research within this doctoral thesis was carried out in the foundry section of the ALRO S.A. company.

Currently, Alro's production is oriented towards the manufacture of high and very high value-added products used in various applications, including high-end fields such as aero, automotive, military, electrical industry, civil construction, etc.

All these areas have as an intrinsic requirement the guarantee of the internal quality of the product, which means strict conditions imposed on the cast semi-finished product regarding the chemical composition, level of inclusions, level of hydrogen or alkaline elements, internal structure, shape and surface conditions, etc., in order to obtain the properties and parameters imposed by the customers for the finished laminated product.

The internal quality of cast products depends not only on the chemical composition and solidification conditions, but also on the content of inclusions or H2, characteristics that have an important influence on their internal structure.

According to specialized literature[4-6], most metals in the liquid state contain various types of oxide, carbide and boron inclusions, and the formation of casting defects is an inevitable consequence of their presence. Inclusions can come from the raw materials used, the refractories, the working environment, the slag formation process, and even the processes or materials used to refine the melts. In order to avoid rejection of the cast product it is important to understand the formation mechanism of each type of inclusion and to apply the most effective melt treatment method.

1.2. Types of inclusions present in aluminum and aluminum alloys.

There are three important characteristics that define metal quality: control of chemical composition, reduction of dissolved gases, and removal of non-metallic inclusions. Inclusions in aluminum alloys act as a stress-increasing factor and can cause cracking during or at the end of casting, and also machinability and obtaining the desired physico-mechanical characteristics. [1] [4-6]

The inclusion is defined as the particle or agglomerate, with chemical composition, shape and morphology varied but different from that of the base metal or alloy. These particles or agglomerates can be metallic and non-metallic constituents, in solid or liquid state. Their size can vary from 1 µm to 100 µm, and in the case of agglomerations it can reach tens of millimeters. Inclusions represent separate phases of particles or agglomerations (clusters), which are not compactly bound to the rest of the melt and which appear in the microstructure of aluminum and cast aluminum alloys.

The inclusions arise from the process of obtaining primary aluminum and from the elaboration of the metal melt, thus:

• the formation of non-metallic inclusions depends only on the presence of oxygen or an oxygen-carrying agent (eg: ambient humidity);

• the formation of metallic or intermetallic inclusions is strongly dependent on the chemical composition of the melt and the process temperature.

It has been observed from metallurgical practice that any inclusion larger than 10 µm is harmful to the cast product, and inclusions smaller than 10 µm, if present in large quantities, can cause major defects in aluminum castings, especially if it has the ability to agglomerate (eg: oxides, borons).

The electrolytic aluminum obtained by the Héroult-Hall process has a large number of non-metallic inclusions, with dimensions smaller than 50 µm and approx. 0.5 ppm H2 content. The inclusions are in the form of particles of oxides (Al2O3), spinels (MgAl2O4) and carbides (SiC, Al4C3), with a higher melting point than aluminum. [9]

CHAPTER 2. METHODS OF REDUCING INCLUSIONS IN THE METALLIC BATH

Over time, various methods have been developed to reduce the level of inclusions in the metal melt:

- fluxing with salts, manually or by injection;
- reactive gas injection;
- gravitational sedimentation, which has a maximum effect on particles with dimensions d>40 µm;flotația, care are efect maxim asupra particulelor cu dimensiuni d>20 - 40 µm;
- filtering, which retains inclusions with dimensions <20 µm, depending on the fineness of the filter used or the complexity of the filter battery.

2.1 Removal of inclusions from the metal melt using salts

Removal of inclusions from the metal bath using salt-based fluxes is common in aluminum foundries. Fluxes are solid salts or mixtures of salts used in the metallurgical industry in the process of melting, refining, modifying or protecting against oxidation. The fluxes used in the melting and liquid treatment of aluminum alloys must have a series of chemical, physical and electrical properties that allow them to play a decisive role on the quality of the finished product.

The salt-based flux can be added to the molten metal manually and mixed with the scraper, by lance injection, or by injection with specialized rotors, using an inert gas as the carrier. [1]

However, it should be noted that, in the case of treatment with salts, since they are liquid at the process temperature, special attention is needed when cleaning the furnace, which must be done with slow movements, to ensure that no remains of salts remain in the melt. [7]

It is recommended that the production flow during elaboration - casting includes as many methods as possible to remove inclusions, i.e.: fluxing - flotation - sedimentation - chlorination - filtration which, although this does not guarantee 100% removal of inclusions, ensures the maximum possible yield .

2.2 Sedimentation/flotation of inclusions in the metal bath

Regarding the process of sedimentation - flotation of inclusions from the metal melt, the following aspects should be highlighted:

• the specific segregation of some of the inclusions is not strictly the result of the gravitational force, but also the result of the direction of the metal flow during the tilting and emptying of the furnace;

• depending on the density compared to the metal bath and the size of the inclusions, the segregation is progressive; inclusions with a higher density than the metal bath settle to the hearth of the furnace, while inclusions with a lower density than the metal bath float/migrate to the surface;

• sedimentation/flotation decreases to zero if the difference in specific gravity between the metal bath and inclusion particles is small;

• in the case of inclusions with the lowest densities (700 – 1800 kg / m3), their size does not play a significant role regarding their direction of movement, but, as a rule, they remain trapped in the metal bath;

• the size of the inclusions is very important for inclusions with higher density than the melt. These large inclusions (d ≥20 μm) settle heavily on the hearth of the furnace during settling, but when emptying the furnace, due to the movement of liquid metal fronts, their concentration becomes higher in the zone of emptying the furnace, moreover, their concentration increases towards the end of the casting. [7]

With the help of tests [20], the time required for sedimentation/flotation was calculated and it was highlighted that:

• for particles with dimensions d>20 μm, the time required for sedimentation is approximately 30 minutes;

• particles with dimensions 10<d<20 μm need additional time for sedimentation, but regardless of the time, they do not sediment in large quantities;

• particles with dimensions d<10 μm remain in suspension in the metal bath regardless of the waiting time;

• increasing the waiting time, from 30 minutes to 60 minutes, has no significant effect on the sedimentation of particles with sizes d≤20 µm.

Therefore, in the usual processing practice, in the furnace, the sedimentation of particles with dimensions d>20 µm is observed, for which a waiting time of approximately 30 minutes is necessary, and particles with dimensions d<20 µm will be reduced/eliminated by other specific operations (ex: filtering).

2.3 Filtering

Filtration, like yield, is the most effective step in removing inclusions, even those dissolved in the aluminum melt. The removal of inclusions by filtration greatly improves the mechanical properties of the products, but filtration, as a single operation, does not ensure the internal quality required by cast products.

The filtering efficiency [22] is defined as follows:

$$E = C_{in} - C_{out} / C_{in} \qquad \{14\}$$

were: c_{in} = level of inclusions before entering the filter,

c_{out} = level of inclusions after exiting the filter

The efficiency of the filtration depends directly on:

• the initial level of inclusions in the melt;

• the size of the inclusion particles: the larger the inclusion particles are, the more effective the filtration, but if they are too large and too many they can block the filter and it can damage under its own weight leaving, further on in the process of casting, so that the inclusions pass freely and end up in the cast product.

• filter pore fineness: the finer the filter, the smaller the size of the particles of inclusions captured.

On a large scale, in aluminum foundries, "Ceramic Foam Filter" (CFF), open pore filters, are used, which are manufactured by impregnating, at granular level and under high temperature conditions, polyurethane foam with ceramic suspension, process which ensures cohesion between the two materials, resulting in a porous body with an open pore structure. Ceramic filters can be placed in filter boxes, vertically or horizontally, and are available in different dimensional shapes and different pore sizes: between (20 – 550) pores/inch, which means a pore size of 0.3 – 0.6 mm. CFF is a rigid and easy to handle, technologically advanced filter.

It is observed that the filtration efficiency, [E] – relation 14, increases directly proportional to the size of the inclusion particles. Thus:

• E >1, there is a small number of particles with dimensions > 60µm, particles that are captured by filtration;

• E= 0.4 – 0.5 there is the largest number of particles that are captured by the filter, with dimensions (60-20) µm, for these filter particles having the highest efficiency. Of this size, approx. 85% of the particles that hit the filter;

• E< 0.3, for particles with sizes <20 µm, the filtration efficiency is not remarkable.

For the reasons stated above, the amount, size and type of inclusions that reach the filtration system are very important. These inclusions that reach the filter system obviously come largely from the metal bath in the melting/making furnace, therefore the treatment of the metal bath in the furnace and the sedimentation/flotation time of the metal bath are vital to avoid filter clogging, ensure filtration efficiency and improve the internal quality of the cast product.

PART II

PERSONAL INDUSTRIAL EXPERIMENTAL RESEARCHES ON THE EVOLUTION OF THE LEVEL OF INCLUSIONS IN ALUMINUM AND ITS ALLOYS DEPENDING ON PROCESSING AND CASTING CONDITIONS

CHAPTER 4. INDUSTRIAL EXPERIMENTAL RESEARCH ON THE PRODUCTION FLOW

4.1.The evolution of the level of inclusions following the treatment of the metal bath in the furnace with different types and amounts of flux

A series of tests were carried out on 5754 and 5083 alloys (the chemical composition is shown in table 1), with different fluxing methods, type and quantities of flux used in the treatment of the metallic bath.

Table 1 **Chemical composition of 5754 and 5083 alloys**

Alloy	Si %	Fe %	Cu %	Mn %	Mg %	Cr %	Zn %	Ti %	Other		Obs.	Al %
									Each	Total		
5083	0.4	0.4	0.10	0.4-1.0	4.0-4.9	0.05-0.25	0.25	0.15	0.05	0.15	-	Bal.
5754	0.4	0.4	0.10	0.50	2.6-3.6	0.30	0.20	0.15	0.05	0.15	Mn+Cr = 0.10-0.6	Bal.

The tests were carried out in the Foundry Department of ALRO S.A., the alloys were produced in gas furnaces with a capacity of 60 tone, and the casting was carried out on an installation using Wagstaff technology.

Depending on the type of alloy and the application of the cast product, on the production flux of the slabs from the Alro Foundry, the removal of inclusions from the metallic melt is carried out by dragging with salts, in one or two stages of treatment, as follows:

- one stage: manual, treatment with drossing flux;
- two stages: manual, with drossing flux + manual, with Na/Ca reduction flux;
- two stages: manual, with drossing flux + lance injection (inert gas = Ar), with Na/Ca reduction flux.

For the tests, it was used:

- the Na/ Ca reduction flux supplier A

- drossing flux, supplier B,

- Na/ Ca reduction flux, supplier C.

According to the technical sheets sent by the suppliers, the technical data of the fluxes used are presented in Table 2:

Table 2 . **Characteristics of the fluxes used for experiments**

Flux	**Amount** [Kg/ t]	**Temperatures** [ºC]
Flux A	1 – 1.5	min. 700
Flux B	1 – 1.5	min. 700
Flux C	min. 0,5 kg/t	min. 480

After the treatment with salts, the slag resulting from the flotation process is extracted from the surface of the metallic bath. If the metallic bath has the chemical composition according to the customer's specification, for the decantation of large and heavy inclusions and for the flotation of inclusions with a specific weight lower than the melt, it is left for at least 30 minutes without interfering with it.

It should be conclude that:

- the PoDFA (Porous Disc Filtration Apparatus) samples for measuring the level of inclusions were taken at a length of the batch between 2000 – 2500 mm. Since the PoDFA analysis method is a pointed method and the movement of the inclusions during the rocking of the furnace, depending on their density and size, is random, the level of inclusions is variable depending on the casting length (degree of inclination of the furnace).

According to the literature, in the case of large-sized inclusions (≥20 μm) that settle on the bottom of the furnace, during the rocking of the furnace, they migrate to the evacuation zone, which leads to a significant increase in their level towards the end of casting;

- in all cases the sedimentation time was 30 minute, to ensure the sedimentation of large inclusions (d≥20μm);

- in all cases the temperature was 718ºC ≤ T≥ 735 ºC, above the temperature recommended by flux suppliers (Table 2), thus fulfilling the recommendation from the literature regarding ensuring the reactivity of the metal bath - flux.

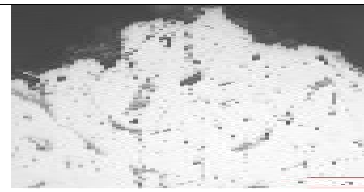
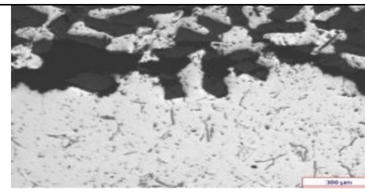
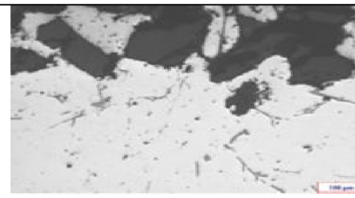
Following the metallographic analysis on the collected PoDFA samples, the existence of inclusions, especially oxides - film of oxides, spinel, MgO, carbides and borons, but also flux particles - in the case of some brazes (alloy 5083) was highlighted.

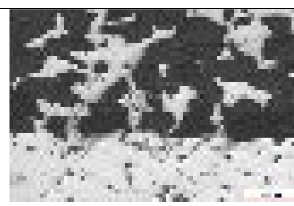
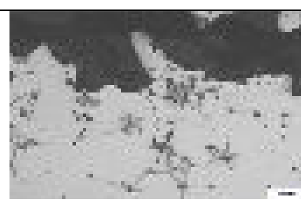
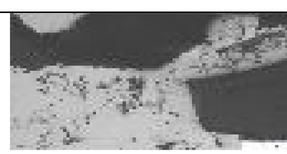
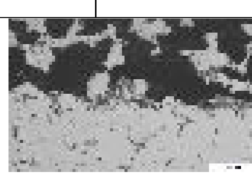
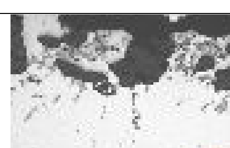
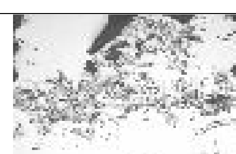
The results obtained from the metallographic analysis of the PoDFA samples are presented in Table 3.

Table 3.

The results of the metallographic analysis of the Podfa sample, taken from the furnace after the flux treatment

Alloy	**Batch**	**Metalographic analysis PoDFA**

5754	1.1	
	1.2	
	1.3	
	The main inclusions identified are: oxides (film), spinels, MgO, carbides, borons	

Alloy	Batch	Metalographic analysis PoDFA	
5083	2.1		The identified inclusions are: oxides (film), spinels, MgO, carbides, boride
	2.2		
	2.3		
			

	For batch 2.2, in addition, agglomerated flux particles were identified, also highlighted by the high value of the level of inclusions = 1.792 [mm^2/kg]

5754 Alloy

PoDFA samples from three cast batches from 5754 alloy were taken and analyzed, according to Table 4.

Regarding alloy 5754, the following can be observed in figure 1:

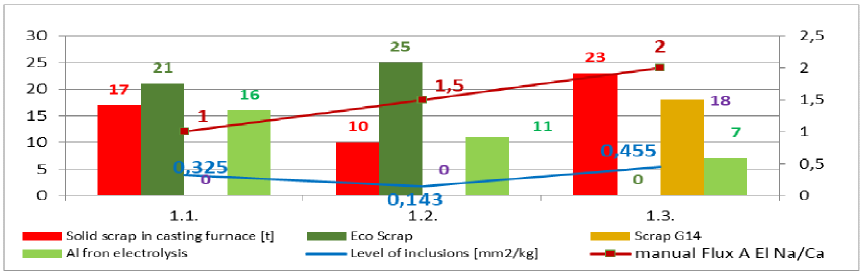


Fig. 1. The level of inclusions depending on the amount of flux used in 5754 alloy

- for all the three batches, was used waste (scraps), both melted directly in the processing furnace and previously melted in a separate furnace;
- the same drossing flux type A Na/Ca was used, the same method of fluxing the metal bath - manual fluxing - and the amount of treatment flux was varied;
- increasing the quantity of zurification flux above 1.5 kg/t metallic bath does not seem to help to reduce the level of inclusions. It can be observed that by increasing the amount of flux to q=2 kg/t liquid, the largest amount of inclusions was measured (batch 1.3, level of inclusions =0.455 [mm^2/kg]);
- the amount of electrolytic metal seems to have a random influence on the level of inclusions, batches in which the amount of electrolytic metal was higher have a high level of inclusions, compared to batches in which the amount of electrolytic metal was lower;
- the level of inclusions seems to be significantly influenced by the amount of solid waste fed directly into the processing furnace. It can be assumed that treating with flux and extracting the slag from the liquid metal coming from the melting of the waste beforehand in another furnace before being charged in the elaboration furnace reduces the influence of this type of metal on the final level of inclusions;

in the case of alloy 5754, the flux treatment of the metal bath, quantity q=1.5 kg/t using the manual method (Flux A +drossing flux Na/Ca) gave the best results, in the conditions where solid waste was loaded including in the casting furnace.

Table 4

Level of inclusions in the furnace after treating the melt, varying the quantities, methods and types of flux

Alloy	Batch	Flux				Q scarp [t]				Al liq [t]	Q total [t]	Inclusion level [mm^2/kg]
		Method	Q [kg/t]	Total flux [kg]	Flux type	solid	Eco	G 14	Total			
	1.1	manual	1	1	Flux A +drossing	17	21	0	38	16	54	

					flux Na/Ca							**0.325**
5754	1.2	manual	1.5	1.5	Flux A +drossing flux Na/Ca	10	25	0	35	11	46	**0.143**
	1.3	manual	2	2	Flux A +drossing flux Na/Ca	23	0	18	41	7	48	**0.455**

Alloy 5083

PoDFA samples from 3 cast batches from 5083 alloy were taken and analyzed, according to Table 5.

*Table 5***Level of inclusions in the furnace after treating the melt, varying the quantities and methods and types of flow**

Alloy	**Batch**	**Flux**						**Q scrap [t]**				**Al liq [t]**	**Q total [t]**	**Inclusion level [mm²/kg]**
		Method 1	**Q [kg/t]**	**Method 2**	**Q [kg/t]**	**Total flux [kg]**	**Type of flux**	**solid**	**Eco**	**G14**	**total**			
5083	2.1	manual	1	manual	0.5	1.5	Flux B + Flux C	15	0	25	40	7.4	47.4	**0.372**
	2.2	manual	1	lance	0.5	1.5	Flux B + Flux C	13	14.1	0	27.1	21	48.1	**0.300**
	2.3	manual	1	manual	1	2	Flux C	10	14	8	32	14.5	46.5	**1,792**

Regarding alloy 5083, the following can be observed (figure 2):

- for the batches analyzed, liquefied metal from the Eco Smelter, waste melting furnace and Electrolliza was used;
- for all the batches studied, waste melted directly in the casting furnace was used -for processing;
- as in the other cases studied, the most significant influence on the level of inclusions seems to be the solid waste fed directly into the processing furnace. It can be assumed that, because

the liquid metal from the waste melting in the G14 furnace is treated with flux and the slag is extracted before being fed into the processing furnace, its influence on the final level of inclusions is reduced;

- for the studied batches, two types of fluxes were used (type B drossing flux and type C – Na/Ca reduction flux), both fluxing methods (manual and lance) and the amount of flux used, both total and on the fluxing stage;

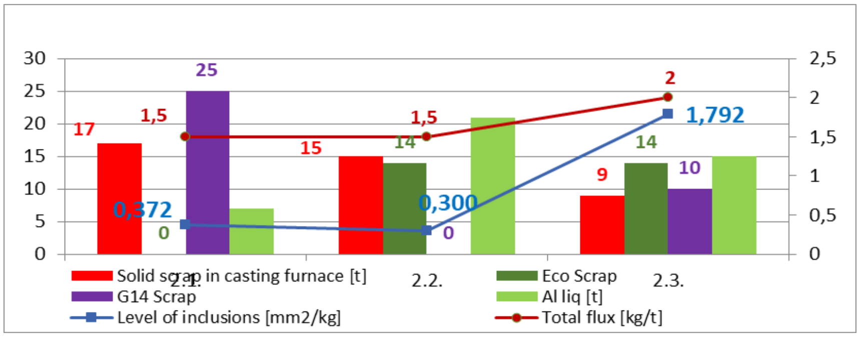


Fig. 2. The level of inclusions depending on the amount of flux used in 5083 alloy

Regarding the type of flow, the quantity and the method used, in figure 3 the following aspects can be observed:

- for the cases studied, the increase in the total amount of flow has a random influence. It can be observed that:

• at the same amount of flow q=1.5 kg/t, using two types of flow, the results are similar, inconclusive;

• using the same amount of flow and two different methods, the results are similar, inconclusive;

- if the amount of flux increased and the total level of inclusions increased, flux particles remaining in suspension in the metal bath were identified. However, it is not possible to correlate the increase in the level of inclusions with the amount of flow used, so it cannot be established that by reducing/increasing the amount of flow, a reduction/increase in the level of inclusions will be obtained.

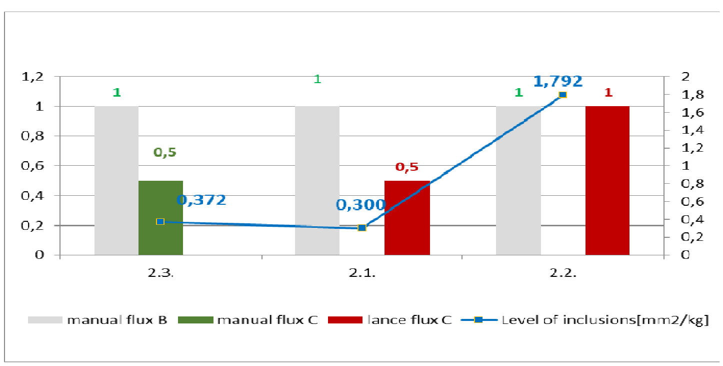


Fig. 3. The level of inclusions depending on the amount of flux and the fluxing method in 5083 alloy

Once again, it is worth mentioning the attention that must be given to the complete elimination of flux salts from the metallic bath - the influence of the human factor.

Therefore, the effect of reducing or increasing the amount of flow, considering the test results, was inconclusive.

The level of inclusions in the metallic aluminum bath is substantial, their size and type is varied. Reducing the level of inclusions represents a challenge for aluminum foundries, the result being directly dependent on several factors and parameters that intervene in the process, the most important being:

- quality of raw materials and auxiliary materials;

- process parameters, especially temperature;

-working method;

- the human factor.

According to literature, for molded deformable products intended for lamination for general use, the recommended level of inclusions = 0.01 mm2/kg, under the conditions that the level of H_2 < 0.1 ml/100 g liquid. In the case of cast products with special destinations, both the level of inclusions and hydrogen are recommended to be even lower.

Fluxing the metal bath, sedimentation and extraction of slag is a critical operation, the results obtained depend on many variables. The tests showed that if a similar amount of flux and a similar method of fluxing are maintained, the results obtained evolve randomly, in a large range of values.

The tests carried out in the ALRO Foundry regarding the influence of the quantity and type (commercial brand) of salt flux used on the reduction of the level of inclusions in the metal bath did not give conclusive results. Furthermore, the laboratory analyzes showed that slags with a low level of inclusions developed, in the cast product, oxide in the network of considerable sizes and porosity of dimensions comparable to slags with a much higher level of inclusions.

It could not be established with certainty that the type of flow used influenced the increase/decrease of the level of inclusions, the results being random.

For the cases studied, the increase/decrease of the total amount of flux shows a random influence on the level of inclusions. Analyzing the results of the PoDFA analysis shows that the level of inclusions after flux treatment is not directly dependent on the amount and type of flux used, but, according to the literature [5-8], on the level of reactivity and the dimensions of the components/inclusions in the metallic bath at the temperature the process, in particular, the density/type of particles that form the inclusions and the way of working. If the density of the particles is close to the density of the metal bath or if the working parameters for fluxing and slag extraction (temperature) are not respected, the flow has a reduced capacity to transport them so that most of them remain in suspension in the metal bath, more , remain in suspension and moistened flow particles.

The tests seem to demonstrate that the treatment of the metal bath with the help of the lance is not the most appropriate choice, a situation also presented in other specialized studies. The flow blowing treatment with the lance causes strong turbulence, which can lead to the formation of bifilms of oxides that are much more difficult to remove from the melt, and which will be found as inclusions in the solidified product.

The high level of inclusions measured for certain tested batches probably has a consistent connection with the flux particles remaining in suspension in the metal bath and highlighted by the metallographic analysis. In the case of these bales, the treatment was the usual one (1.5 kg/t flow, method: manual + lance). Because during the treatment with the lance the temperature of the bath decreases and considering that the low temperature of the process is the factor with the greatest influence in the case of keeping these particles in suspension in the metallic bath (+ the human factor), it can be stated that the treatment with the lance adds a additional problem in the process.

Reducing or increasing the amount of flux compared to the amount recommended by the manufacturer or the type of the flux does not guarantee a reduction in the level of inclusions. For this reason there is no international standard to regulate this operation, the Aluminum Association's recommendation is testing, on the production flow, both the type and the required amount of fluxes, starting from the supplier's recommendations and adapting them to the production needs.

4.3 The influence of filtration on the level of aluminum inclusions and aluminum alloys

During the experiments, on the route of the metal transfer troughs from the tallow casting plant, after the degassing pot, an additional filter box was installed which, together with the already existing one, would ensure a two-stage filtration system.

The standard chemical composition, according to SR-EN 573-3, of the three alloys is shown in table 6.

Table 6

Standard chemical composition of the experimental alloys

Alloy	Si, %	Fe, %	Cu, %	Mn, %	Mg, %	Cr, %	Zn, %	Ti, %	Other		Al
									Each	Tot al	
5083	0.40	0.40	0,10	0.40-1.0	4.0-4.9	0.05-0.25	0,25	0,15	0,05	0,15	bal
6061	0.40-0.8	0.70	0.15-0.40	0,15	0.8-1.2	0.04-0.35	0,25	0,15	0,05	0,15	bal
7075	0.40	0.50	1.2-2.0	0,30	2.1-2.9	0.18-0.28	5.1-6.1	0,20	0,05	0,15	bal.

The technological flow diagram of the filtration process of aluminum alloys is presented in figure 4.

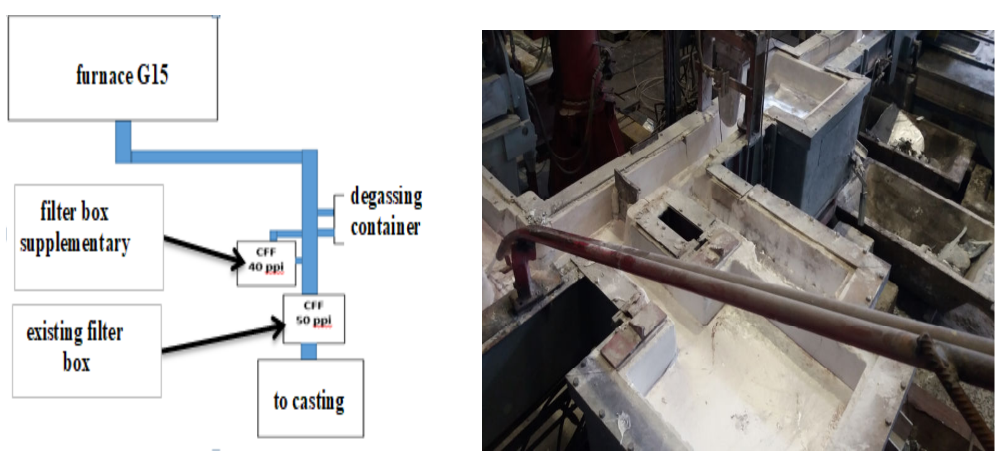
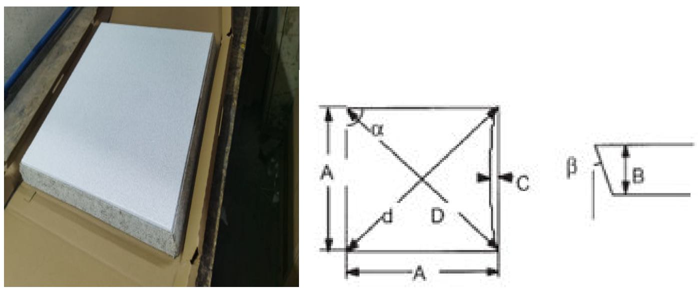


Fig. 4. Schematic of the technological flow of filtration process a) with two-stage filtration in aluminum slab casting plant, b) image of the two-stage filtration system in an slab casting plant

Filtration of the liquid metal flow is carried out in an in-line filtration system, a single filtration stage using a filter 584 x 584 mm (23 inch) and 40 ppi porosity. Ceramic foam filters are used worldwide to filter molten aluminum. It is an alumina-based, high-purity, phosphate-bound product whose characteristics can be found in figure 5.

Ceramic foam filters (CFF) were used for the tests as follows:

- stage 1 (newly installed filter box) – CFF 40 ppi filter, in all cases.
- stage 2 (existing filter box) - CFF filter 50/60 ppi



FILTER SIZE	DIMENSION A (MM)
7 inch	178 x 178 ± 2
9 inch	229 x 229 ± 2
12 inch	305 x 305 ± 2
15 inch	381 x 381 ± 2
17 inch	432 x 432 ± 3
20 inch	508 x 508 ± 3
23 inch	584 x 584 ± 3
26 inch	660 x 660 ± 4

PROPERTY	SPECIFICATION
angle α	90° ± ½°
angle β	17½° ± ½°
B	50 mm ± 2 mm
bend C	± 1½ mm on each edge
D-d	≤0.6% x A

Fig. 5. Shape and specificiation of CFF filter used for experiments

To determine the level of purity of the metal bath, periodically, samples are taken, which are analyzed in Metallographic Analysis Laboratory. Out of the total of 5 castings proposed to be carried out.

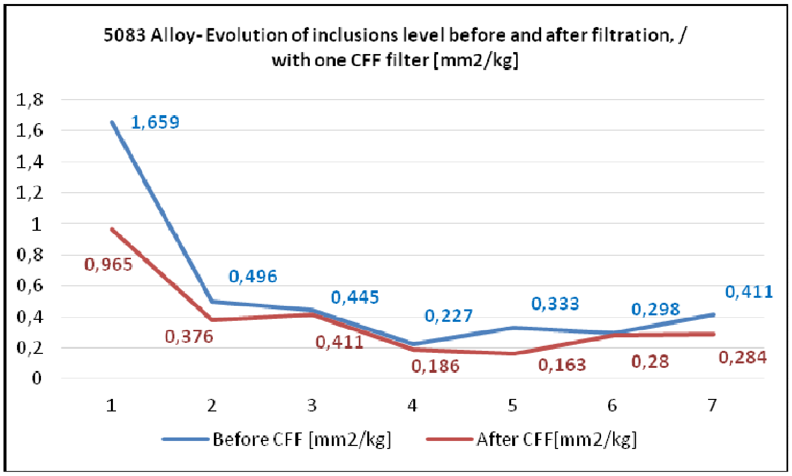
Since the usual recipe used in the casting of these alloys was used, without making any changes, the batches were started in "manual" mode, tilting the furnace as much as the capacity of the transfer chute allowed, ensuring the approx 800 kg more.

The "Dual Stage" filtering system, installed at the slab casting line, was tested when casting slabs from alloys 5083, 6061, 7075, format 1650 x 500 mm, the CFF configuration used is shown in table7 .

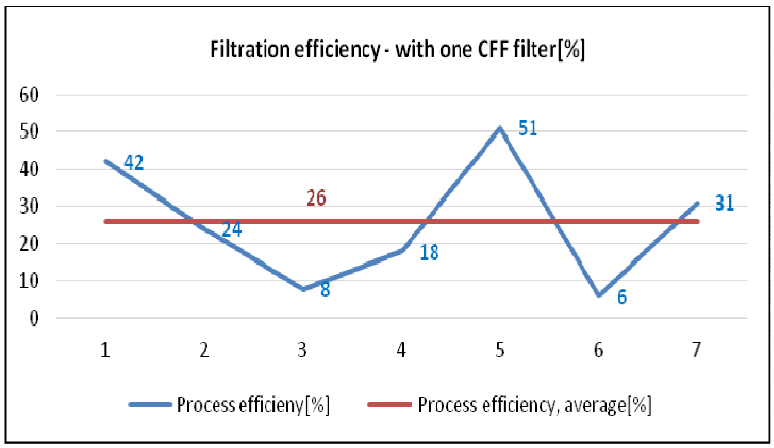
Table 7 **Configuration CFF two stage filtration system and cast alloys**

Two-stage filtration system		**Alloy type**	**No of castin gs**	**Observations**
Stage 1	Stage 2			
CFF 40 ppi	CFF 50/60 ppi	5083	2	At the start of casting, a larger amount of metal is required to prime the additional filter (+approx. 800 kg). The transfer chute has the capacity to take this additional quantity; the operator must start in "manual" operating mode. After starting, the level of metal on the chute balances, the casting proceeds normally, without problems (samples were taken in all cases).
		6061	2	
		7075	1	

The evolution of the level of inclusions in the produced slugs and the efficiency of the process, in the filtration system currently used with a filtration stage using a CFF 40 ppi filter is shown in figure 2 (a and b), for aluminum alloy 5083 and figure 3 (a and b) for aluminum alloy 7075.

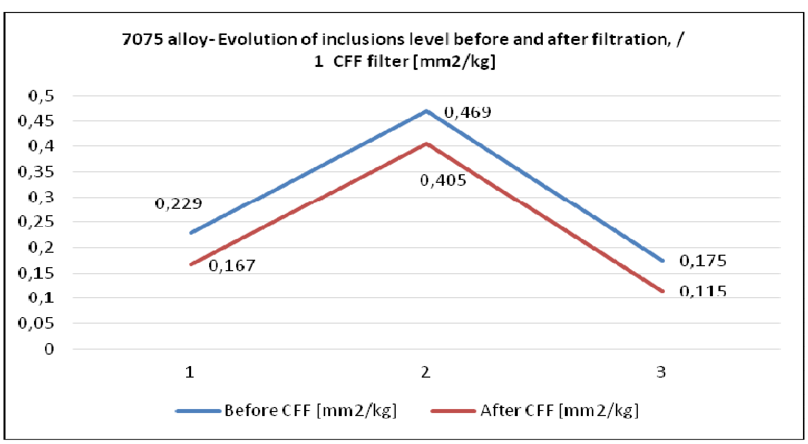


a)

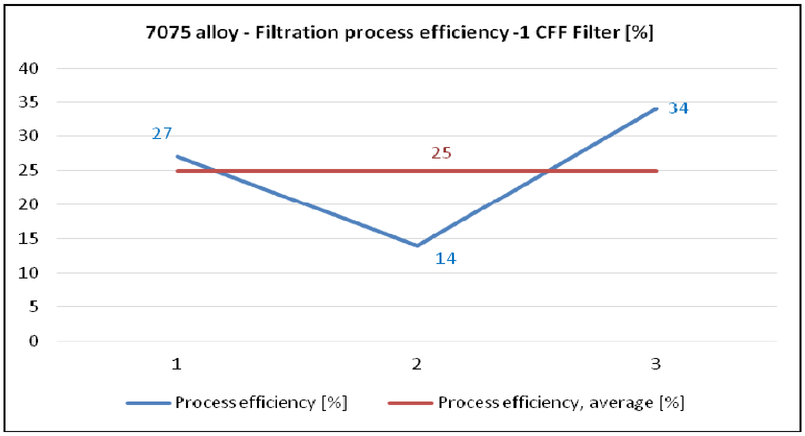


b)

Fig. 6. Alloy 5083 – a) Level of inclusions before and after filtering, CFF filter 40 ppi, b) Filtration process efficiency, alloy 5083



a)



b)

Fig. 7. Alloy 7075 a) Level of inclusions before and after filtering, CFF filter 40 ppi, b) Filtration process efficiency, alloy 7075

The level of inclusions before filtering represents the level of inclusions in the metal bath in the processing furnace, and the level of inclusions after filtering represents the level of inclusions in the cast product. It is observed that both the level of inclusions and the efficiency of the filtration process have a random behavior, and the final result depends, directly proportionally, on the level of inclusions in the metal bath in the furnace, after it has been treated with flux and the slag extracted.

Three samples were taken for each batch as follows:

- a sample when the metal is in the furnace, before the degassing pot;
- a sample after the first filtering stage (CFF 40 ppi filter)
- a sample after the second stage of filtering (CFF filter 50/60 ppi)

The results are presented in table 8.

Table 8.

The results of the analyzes on the experimental samples

Alloy	Batch	Sample		Discussions
		Prelevation zone	Level of inclusions mm^2/kg	
5083	1	furnace	0.931	The evolution of the level of inclusions indicates a good filtration of the metal, the total efficiency of filtration = 88% (stage 1 = 56%, stage 2 = 73%)
		after filter 40 ppi	0.406	
		after filter 50 ppi	0.107	
	2	furnace	1.999	The evolution of the level of inclusions indicates a good filtration of the metal, the total efficiency of filtration 93% (stage 1 = 87 %, stage 2 = 49%)
		after filter 40 ppi	0.263	
		after filter 60 ppi	0.135	
6061	3	furnace	0.122	The evolution of the level of inclusions indicates a good filtration of the metal, the total efficiency of filtration = 68% (stage 1 = 13%, stage 2 = 64%)
		after filter 40 ppi	0.106	
		after filter 50 ppi	0.038	
	4	furnace	0.179	The evolution of the level of inclusions indicates a good
		after 40 ppi filter	0.104	

		after 50 ppi filter	0.065	filtration of the metal, the total efficiency of filtration = 64% (stage 1 = 42%, stage 2 = 38%)
7075	5	furnace	0.846	The evolution of the level of inclusions from one filtering stage to another indicates an error in the sampling or traceability of the samples. Efficiency of filtration = 89 % (stage 1 = 84%, stage 2 = 34%)
		after filter 40 ppi	0.135	
		after filter 60 ppi	0.089	

Thus, comparing the total result of filtering in the case of batch 5083 using a single CFF 40 ppi filtering stage (fig. 6 a, b), the maximum filtration efficiency obtained 51% is lower than the result obtained in the case of using two stages of filtering (CFF 40 ppi+ 60 ppi) where the total efficiency of the process is 93%.

Also, comparing the results obtained during the tests after each of the two casting stages, the filtration efficiency obtained after the second filtration stage is higher than the efficiency obtained after the first filtration stage.

The efficiency obtained after each filter stage and the total efficiency are presented in figure 8.

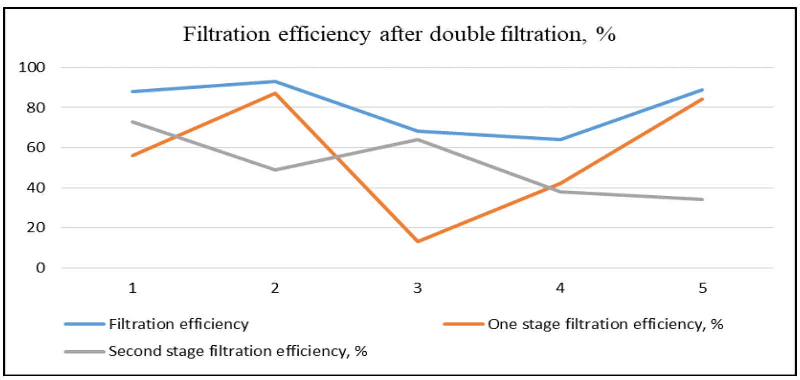


Fig. 8.Double filtration efficiency, %

According to theory, the CFF 40 ppi filter retains inclusions with size >20µm and the CFF filter (50/60 ppi) retains inclusions with size <20µm.

It is obvious that the high efficiency obtained on the second filtering stage is due to both the retention of inclusions with dimensions <20µm and the retention of inclusions with dimensions >20µm, which passed the 40 ppi filter (stage 1)

The addition of the second filtration stage to the production flow from the foundry section involves additional costs, both in relation to the second CFF filter and in relation to the purchase of a high-performance filter box for the second filtration stage, to avoid drastic temperature drops at start-up and to reduce the risk of rejecting the charge.

The current liquid metal filtration system can be improved by adding an additional filter box and testing the efficiency of the filtration installation in two stages, using CFF 40 ppi filters, in stage 1 and CFF 50/60 ppi filter (or other porosity, depending of the destination of the finished product), in stage 2.

On the test samples whose results can be taken into consideration, comparing the result obtained after two filtration stages (CFF 40+50/60ppi) with the results obtained using one filtration stage (CFF 40 ppi) it can conclude:

- in the case of alloy 5083, using one filtration stage (CCF 40 ppi), the filtration efficiency is 56% is lower than the result obtained in the case of using two filtration stages (CFF 40 ppi+ 60 ppi), where the total efficiency of the process is 88 %.

- in the case of alloy 6061, using one filtration stage (CCF 40 ppi), the efficiency = 13% is lower than the total efficiency of the process = 68%, obtained in the case of using two filtration stages (CFF 40 ppi+ 50 ppi).

The efficiency values presented above show that, by using a two-stage filtration system, an increase in the efficiency of removing inclusions from the liquid metal can be obtained.

CHAPTER 5. FINAL CONCLUSIONS, ORIGINAL CONTRIBUTIONS AND FUTURE RESEARCH DIRECTIONS

5.1. Final summary conclusions

5.1.1. The level of inclusions in the aluminum metal bath is substantial, their size and type being varied. Reducing the level of inclusions is a challenge for aluminum foundries, the result being directly dependent on several factors and parameters that intervene in the process, the most important being:

- quality of raw materials and auxiliary materials;
- process parameters, especially temperature;
- working method;
- the human factor.

5.1.2. According to the specialized literature [21], for the deformable cast products intended for lamination for general use, the recommended level of inclusions is 0.01 mm2/kg, provided that the H2 content is below 0.1 ml/100 g Al liq. In the case of cast products with special destinations, both the level of inclusions and the content of H2 are recommended to be as low as possible.

5.1.3. Metal bath fluxing, sedimentation and slag extraction are critical operations, the results obtained depending on many variables.

5.1.4. For the studied cases, increasing/decreasing the total amount of flow shows a random influence on the level of inclusions. Analysis of the PoDFA results shows that the level of inclusions after the flux treatment is not directly dependent on the amount and type of flux used, but on the level of reactivity and the dimensions of the components/inclusions in the metal bath at the process temperature, in particular, the density, the type of particles which form the inclusions and the way of working. If the density of the particles is close to the density of the metal bath or if the working parameters for fluxing and slag extraction are not respected (especially the working temperature), the flux has little capacity to transport their inclusions, so that most of them remain in suspension in the metal bath, more, remaining in suspension and moistened flux particles.

5.1.5. Tests have shown that the treatment of the metal bath with the help of a lance is not the most suitable choice. In the case of tests for alloy 7075, the lowest level of inclusions was obtained under the conditions of treating the metal bath with 1 kg/t using only the manual method (no lance). The high level of inclusions measured for certain batches tested has a consistent relationship with flux particles remaining in suspension in the metal bath and evidenced by metallographic analysis. In the case of these batches, the treatment was 1.5 kg flux/t, the method used being the combined method: manual + lance. Since during lance treatment the temperature of the bath decreases, and considering that the low process temperature is the most influential factor in maintaining these particles in suspension in the metal bath, it can be stated that lance treatment adds an additional problem in trial. In addition, due to the method of treatment with the lance (blowing the flux with the help of an inert gas), on the surface of the metal melt in the furnace, strong turbulences occur, which drive the formed oxide film inside the melt, leading to the introduction of metal inclusions into the bath bifilm type, very difficult to remove in the later stages of the process.

5.1.6. Reducing or increasing the amount of flux used compared to the amount recommended by the manufacturer or the type of flux used does not guarantee a reduction in the level of inclusions. For this reason there is no international standard to regulate this operation, the recommendation of the Aluminum Association being to test, on the production flow, both the type and the required amount of flow, starting from the supplier's recommendations and adapting them to the needs in practice.

5.1.7. The removal of inclusions from the melt is considered a critical operation of the casting process, and none of the existing methods ensure 100% removal of inclusions. For this reason, it is recommended that the casting production flow includes as many methods as possible to remove inclusions, i.e.: fluxing - flotation - sedimentation - chlorination - filtration which, although it does not ensure the complete removal of inclusions, ensures the maximum possible yield .

5.1.8. Sedimentation/flotation are essential technological operations for reducing the level of inclusions in the metal bath with the following observations:

• the segregation of inclusions is not strictly the result of the gravitational force, but also the result of the direction of the metal flow during the tilting and emptying of the furnace;

• depending on the density compared to the metal bath and the size of the inclusions, the segregation is progressive;

• inclusions with a density higher than that of the metal bath settle towards the hearth of the furnace, while inclusions with a density lower than that of the metal bath migrate towards the surface;

• sedimentation/flotation decreases towards zero if the difference in specific gravity is small;

• in the case of inclusions with the lowest densities (700kg/m^3 and 1800kg/m^3), their size is not a significant factor regarding their direction of movement, mostly remaining trapped in the metal bath;

5.1.9. The coating sedimentation time in the case of aluminum alloys is a minimum of 30 minutes, because:

- for particles larger than 20 µm, the time required for sedimentation is approximately 30 minutes. Increasing the waiting time from 30 minutes to 60 minutes has no significant effect on the sedimentation of these particles;
- for particles with sizes between 10µm and 20µm, additional time is needed for sedimentation, but even under these conditions, the efficiency of the process is not sufficient to justify the reduction in productivity. These inclusions are retained, with greater efficiency, in the filtration system;
- particles smaller than 10 µm remain in suspension in the metal bath regardless of the waiting time.

5.1.10. There is a dependence between the level of inclusions in the metal bath in the furnace and the amount of electrolytic aluminum used: regardless of the length of the sedimentation time, if the amount of electrolytic aluminum increases, the level of inclusions also increases. Probably electrolytic aluminum contains inclusions of small size and/or specific gravity approximately equal to the metal bath in the furnace, inclusions that do not sediment/float but remain trapped in the melt.

5.1.11. An analysis of the filtration yield can give an indication of the size and nature/specific weight of the inclusions remaining in the process. In the case of tests carried out:

• no matter how many filtration stages or what type/fineness of filter is chosen, there are limitations of the filtration process, the filtration yield not being able to ensure a 100% reduction of inclusions. In the case of the analyzed tests, the highest yield obtained, after two filtering stages (40+60 ppi), is 93%;

• according to the results obtained, the inclusions that pass the filter system are particles with sizes between 20μm and 25μm;

5.1.12. The liquid aluminum filtration system in the casting process can be substantially improved by adding an additional filter box, using 40ppi CFF filters in the first filtration stage and 60ppi CFF filter (or other fineness, depending on the destination of the finished product) , in the second stage of filtering.

The general conclusion is that, although inclusions cannot be eliminated 100% from the system, it is imperative to limit them by performing with great care (the human factor being decisive in this equation) all the operations that lead to reducing their level: fluxing - cleaning the metal surface from the furnace – sedimentation/flotation – degassing – filtration.

5.2. Original personal contributions

The research undertaken in the framework of the doctoral thesis was aimed at establishing technologies for improving the quality of aluminum cast products and its alloys, as well as increasing the performance of the casting technological flow operations, by developing high efficiency systems, reducing the level of inclusions in the final product being one of the methods that can lead to achieving this goal.

The reduction of the content of inclusions in the metal bath in the furnace is achieved by using fluxes – solid salts or mixtures of solid salts with the help of which the inclusions are transported to the surface of the metal bath to be removed. At the same time on the production flow, in reducing the level of inclusions, a very important role is played by degassing, refining (by adding a refiner) and filtering the metal.

In order to fulfill the objective of the doctoral thesis, during the doctoral internship I carried out scientific documentation activities, laboratory and industrial experiments, I collaborated with researchers and specialists from ALRO S A Slatina, from the POLITEHNICA University in Bucharest, through the CEMS Research Center, with specialists from the metallurgical sector, the industry of metals and non-ferrous alloys.

The original, own contributions made within the doctoral thesis are supported by the following activities:

1. In the first part of the doctoral thesis, I conducted a documentary study based on the specialized literature regarding the current state in the country and abroad, the types of inclusions present in aluminum alloys, their formation mechanisms, as well as the methods used for their removal from the melt.

2. In the second part of the doctoral thesis, I first carried out a documentary and experimental research on the analysis procedures of raw materials, materials and equipment and machinery used on an industrial scale to obtain aluminum alloy slabs.

We have carried out a presentation of the production flow of slimes in Alro and a description of the equipment and apparatus used in the experimental researches, and we have presented the conclusions of these researches.

3. We carried out industrial experimental research on several phases of the technological process of obtaining aluminum alloy slabs and presented and interpreted the results obtained, highlighting the conclusions of these researches.

4. We carried out experimental research on the evolution of the level of inclusions according to the conditions of elaboration / casting of aluminum alloys. We have demonstrated in experimental research that the addition of the second filter box to the production flow significantly reduces the level of inclusions in the cast product. ALRO currently uses the double filtration system in the development and casting of alloys for the aerospace and defense industries.

Taking into account the activity carried out and the results obtained, I consider that the purpose of the doctoral thesis has been fulfilled.

The main objectives of the thesis were reflected in articles and papers presented at symposia and published in specialized magazines. The research results were presented, discussed and approved at a series of specialized scientific forums

5.3. Future directions of research development from the doctoral thesis

As a result of the worldwide interest in improving the efficiency of degassing in the process of casting aluminum and its alloys, and reducing the harmful impact on the environment, in the last decades unconventional solutions have been developed and tested, solutions involving reduced energy consumption, positive impact on the environment (elimination of the use of chlorine or its compounds as degassing agents) and to improve the efficiency of degassing.

Ultrasonic degassing [43] is based on the use of ultrasonic waves of high frequency and power, to treat large quantities of material in liquid state. These high-frequency, high-power waves are distributed uniformly, by means of a sensor, in the surface or inside the melt, vibrating it and allowing the creation of standing waves so that the whole system is vibrated and agitated completely, which leads to the elimination of bubbles of gas existing in the melt.

High intensity ultrasonic waves are used because they have the ability to induce vibrations capable of generating an oscillating pressure in the aluminum melt. In this way, in the regions of minimum pressure in the metal melt, small cavities appear in which small gas bubbles are accumulated, bubbles that represent nucleation centers that grow over time. When their size reaches a certain limit, these bubbles break away from the cavities and are transported, by means of ultrasonic waves, to the surface of the melt.

The successful use of the concept at an industrial level brings with it several aspects mentioned below.

- **Advantages**:

- It is a concept developed for the treatment of large volumes/quantities of liquid metal, the beneficial effect being transmitted throughout the metal mass;

- It can be permanently adjusted, in real time (during pouring), depending on the results obtained from the H2 level measurements, so that the product quality can be ensured, thus reducing the scrap percentage and costs.

- Improves the homogeneity of the metal melt and the refiner;

- It has excellent results regarding the reduction of the H2 level, with secondary action on small wettable inclusions, which usually remain in suspension in the melt and are often found in the final product after solidification;

- Improves the internal structure of the final product;

- Improves the quality of the surface of the products;

- It does not contain moving/rotating parts in the degassing system – it does not require graphite rotors, so the system is robust and with minimal expenses;

- Degassing takes place quickly – the bubbles formed are smaller than those formed in the conventional degassing system;

- It is used in conditions of very low flow of argon and without the need for chlorine (unless it is necessary to reduce the alkaline elements);

- Gas bubbles are formed inside the height of molten metal, so no additional oxidation will occur on the surface of the metal, thus avoiding the appearance of oxide film or bifilm inclusions;

- The amount of slag formed is minimal – the surface of the metal has no turbulence that could lead to oxidation;

- The energy consumption is higher, but it compensates with the economy resulting from the processing of a much smaller amount of slag, if it is also applied to the homogenization of the melt in the elaboration furnace.
- Great environmental benefits by reducing gas emissions and the amount of slag formed.

➢ **Disadvantage**

From what has been highlighted so far in the industrial tests, in order to obtain good results, at an industrial level, it is necessary to establish and set the working parameters (frequency) differently, depending on the type of alloy and the volume of the melt subjected to degassing US.

DISSEMINATION OF THE RESULTS FROM THE DOCTORAL THESIS

Published articles

1.V. Șerban-Tănase, M. Ciurdaș, D. A. Necşulescu *Reducing the level of inclusions from the metallic aluminium melting, using a double filter system,* University Politehnica Of Bucharest Scientific Bulletin Series B-Chemistry And Materials Science, acceptată pentru publicare în luna mai 2023, va apare in Volume 85 Issue 3 Page .... Published 2023 , Indexed ...., Accession Number WOS:......................, ISSN1454-2331, Journal Impact Factor ™ (2022) 0,5

2.V. Șerban-Tănase, M. Ciurdaș, D. A. Necşulescu *The influence of the flux treatment on metallic bath concerning the level of inclusions in aluminum alloys* ,University Politehnica Of Bucharest Scientific Bulletin Series B-Chemistry And Materials Science, acceptată pentru publicare în luna iunie 2023, va apare in Volume 85 Issue 3 Page .... Published 2023 , Indexed ...., Accession Number WOS:......................, ISSN1454-2331, Journal Impact Factor ™ (2022) 0,5

Technical-scientific activity

1. Providing technical assistance in the development process - casting of special aluminum alloys in the Foundry Section of ALRO S.A. (decision)

2. Lecturer qualification course in the profession "Nonferrous metallurgist" code NC 7211.2.4., qualification level II (240 hours theory + 480 hours practice), organized by ALRO S.A. during the period 13.11.2020 – 13.04.2021. (decision)

3. Participant in the target group as a doctoral student scholarship funded by the project "Preparation of doctoral students and postdoctoral researchers in order to acquire applied research skills - SMART", financed by POCU based on Contract no. 13530/16.06.2022-SMIS code: 153734 during the period 10.08.2022 - 16.12.2023.

4. Presentation of the technological process and production sections of ALRO S.A. in the capacity of accompanying groups of visitors, on the occasion of the organization of the event "ALRO Open Doors Day" between 29-30.06.2023

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