INVESTIGATIONS OF THE NATURE OF NON-METALLIC INCLUSIONS IN Al-Mg-Mn ALLOYS

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Non-metallic inclusions in the cast slabs of aluminum alloys have detrimental effects both on the mechanical properties and quality of final products. Their presence in large quantities in slabs leads to the appearance of defects as edge cracking, pinholes and foil-breaks. The aim of this paper is to determine both the quantity and the nature of the non-metallic inclusions in Al-Mg-Mn alloys using porous disc filtration analysis (PoDFA) of the melt. It was established that the total non- metallic inclusion content in cast slabs is in the range 0,012-0,024 mm^2/kg. Electron microscopy analysis (SEM, EDS) has revealed the presence of the following basic inclusions: Al_20_3, $MgAl_20_4$ and TiB_2.

Keywords: aluminum alloy, nonmetallic inclusions, Porous Disk Filtration Analysis, SEM, EDS, X-ray element distribution map.

1. Introduction

Al-Mg-Mn aluminum alloys are known for exceptional performance in extreme environments. These alloys are distinguished by particularly good corrosion resistance in seawater, high plasticity and excellent weldability. Some alloys contain additions of titanium and chronium which are designed to refine the structure and improve mechanical characteristics [1]. Inclusion removal from aluminum melts and their alloys is an important melt treatment process. Aluminum is a reactive metal, readily oxidizing when exposed to the atmosphere.

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It forms stable oxides and nonmetallic inclusions during the melting and handling process [2]. The term „inclusion" refers to any type of undissolved foreign material present in cast metal. Inclusions are well known as a common defect in cast structures and normally are obtained during production processes [3].

Slabs are used in rolling, and their quality determines the quality of the rolled product. The presence of inclusions in slabs contributes to rolling problems, such as edge cracking, pinholes, and foil breaks, which affect the economics of slab production adversely [4]. The methods for inclusion assessment based on the pressure filtration or pre-concentration tests are Porous Disc Filtration Analysis (PoDFA) from Alcan and Liquid Aluminum Inclusions Sampler (LAIS) [5].

The porous disc filtration apparatus (PoDFA) technique was introduced as a method of assessing metal cleanliness by Alcan; using this method, the factors controlling the precipitation and sedimentation of non-metallic inclusions (with dimensions between 20 and 50 µm) could be evaluated [6]. The focus of this paper is to study the assessment of AA5083 melt cleanliness using PoDFA method and electron microscopy analysis (SEM, EDS).

2. Experimental procedure

PoDFA technique is becoming increasingly important in the casting of aluminum alloys as purity is of crucial importance for obtaining castings with acceptable content of non-metallic inclusions typically used in shipbuildings, rail cars, vehicle bodies, pressure vessels and mine skips and cages. This technique involves two-different operations: sampling liquid metal followed by its filtration and metallographic preparation and examination of its [1]. The PoDFA (Porous Disc Filtration Appartaus) is the property of Alcan Int. Ltd. and is licensed for manufacturing to ABB Bomem Inc.

Patent pending for melt cleanliness evaluation that provides information on the composition and concentration of the inclusions in molten aluminum in different stages of melt treatment (melting, degassing and filtration).

This technology has also demonstrated its great potential for process optimization, as well as product improvement and to producing high quality castings. However, the results of the PoDFA give no information about the size of detected non-metallic inclusions. Size of inclusions is an important parameter for mechanical properties because large inclusions can act as cracks. PoDFA technique can distinguish inclusions types and differentiate, for instance, between the level of oxides, carbides, borides and spinels present within an individual sample.In PoDFA system includes all the equipment required for sampling in molten aluminum: a PoDFA sampling station, crucible heater, crucibles and filters (Fig.1). The equipment is compact, portable and economical.

a) b)
Fig.1. PoDFA system: a) PoDFA sampling station and crucible heater, b) PoDFA reusable crucible and filters

The slabs of AA5083 alloys were cast through Wagstaff vertical installation.The casting parameters are: rate 45-50 mm/min, water flow rate 650 l/min, temperature 630^0C. Like metallic samples alloys have used AA 5083 alloys (Al-Mg-Mn system) with chemical composition, in wt. %: 4,26 – 4,69 Mg, 0,41 – 063 Mn, 0,18 – 0,42 Fe, 0,07 – 0,26 Si, 0,06 – 012 Cr, 0,004 – 0,036 Ti. [7] A set of 18 samples of liquid alloy (1 – 2 kg) were filtered under controlled conditions using a very fine porosity filter (40 ppi). When the metal is frozen, the filter, along with the residual metal, is cut., mounted and polished before being analyzed under a microscope. Sampling time was typically 4 minutes.

Metallographic analysis residue allows identification and quantification of the types of inclusions present in the sample. Counting method used was GRID method and measured chord length (11,5 – 12,2 mm). The total accumulated values of inclusion concentration area per kilogram was calculated using the formula:

Total Inclusion (mm^2/kg) =[{Mean Measured Residue Area (mm^2) x Inclusion Area Fraction}/{Filtrate Metal Weight (kg)]} x [{ Nominal Chord Length (mm)}/ Measured Chord Length (mm)}] (1)

The polished samples were analysed using a scanning electron microscope (SEM) equipped with an energy – dispersive x-ray spectrometer (EDS).

3. Results and disscusion

It was established that the inclusion content is between 0.012 mm^2/kg and 0.024 mm^2/kg. The chemical composition of AA 5083 alloys and concentrations of non-metallic inclusions are shown in Table 1.

Table 1

Chemical composition of AA 5083 samples (wt. %) and concentration of non-metallic inclusions (mm^2/kg)

Sample	Element [wt%]						Non-metallic inclusion[mm^2/kg]
	Mg	Mn	Fe	Si	Cr	Ti	
1	4,36	0,54	0,30	0,07	0,07	0,014	0,020
2	4,38	0,45	0,33	0,12	0,08	0,015	0,014
3	4,26	0,63	0,31	0,14	0,11	0,004	0,019
4	4,29	0,52	0,34	0,11	0,12	0,016	0,021
5	4,67	0,46	0,33	0,24	0,09	0,012	0,012
6	4,29	0,42	0,42	0,16	0,08	0,013	0,017
7	4,69	0,1	0,25	0,26	0,14	0,031	0,022
8	4,55	0,53	0,27	0,21	0,08	0,026	0,023
9	4,51	0,59	0,26	0,14	0,09	0,034	0,015
10	4,60	0,52	0,29	0,13	0,11	0,028	0,015
11	4,37	0,55	0,25	0,12	0,08	0,034	0,024
12	4,71	0,56	0,19	0,09	0,09	0,025	0,022
13	4,34	0,59	0,22	0,16	0,07	0,036	0,023
14	4,52	0,54	0,24	0,15	0,07	0,031	0,018
15	4,48	0,56	0,36	0,14	0,10	0,033	0,020
16	4,51	0,55	0,15	0,08	0,09	0,036	0,024
17	4,57	0,49	0,98	0,07	0,08	0,032	0,019
18	4,68	0,61	0,26	0,31	0,06	0,020	0,018

SEM metallographic analysis consists of examining the residue of unfiltered metal at the surface of the PoDFA filter. The results of SEM and energy dispersive X-rays analysis of sample 1, are done in Fig.. 2 and Fig.. 3. Very similar alumina inclusions were identified in Samples 2 to 10. The inclusions were located at the cut edges of the mounted samples. The dominant alumina (Al_2O_3) inclusions were considerable large in size (80-100 µm in lengts) and were uniformly distributed across the mounted sample. The alumina inclusions in all of samples have a dense glassy-like microstructure with fracture facets on their surfaces. Possibly sources for the alumina inclusions could be refractory material or undissolved alumina from the pot line **(**reduction cell). It is highly unlikely that these particles would have passed through a 40 ppi ceramic foam filter (CFC) so we would expect the filter to plug during casting.

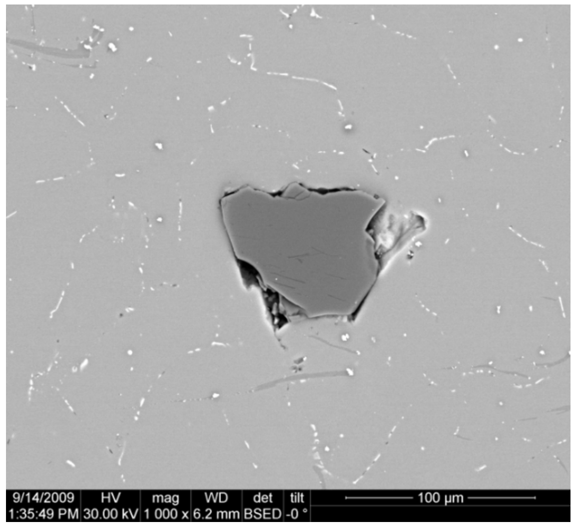

Fig.2. SEM analysis of Sample 1

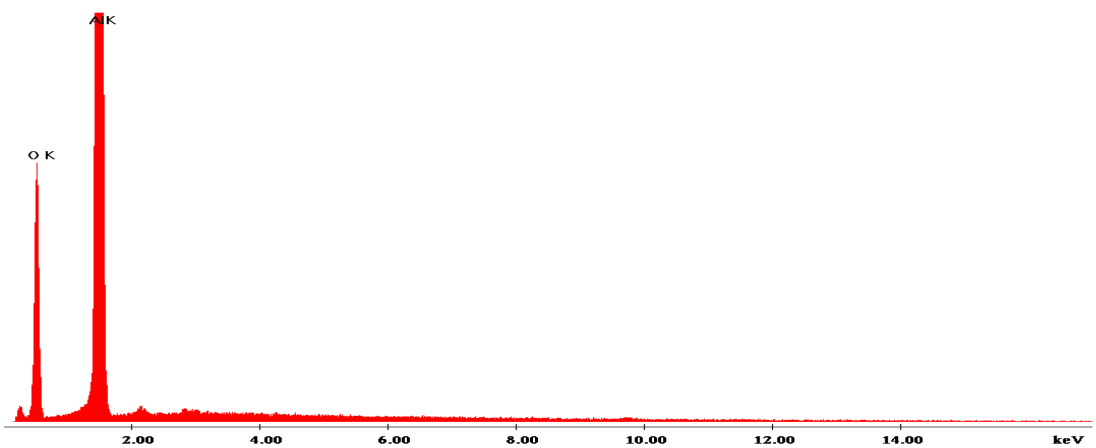

Fig.3 EDS microanalysis of the inclusions in Fig. 2

Fig. 4 present EDS element mapping of an Al_2O_3 particle in the microarea of Fig. 2.

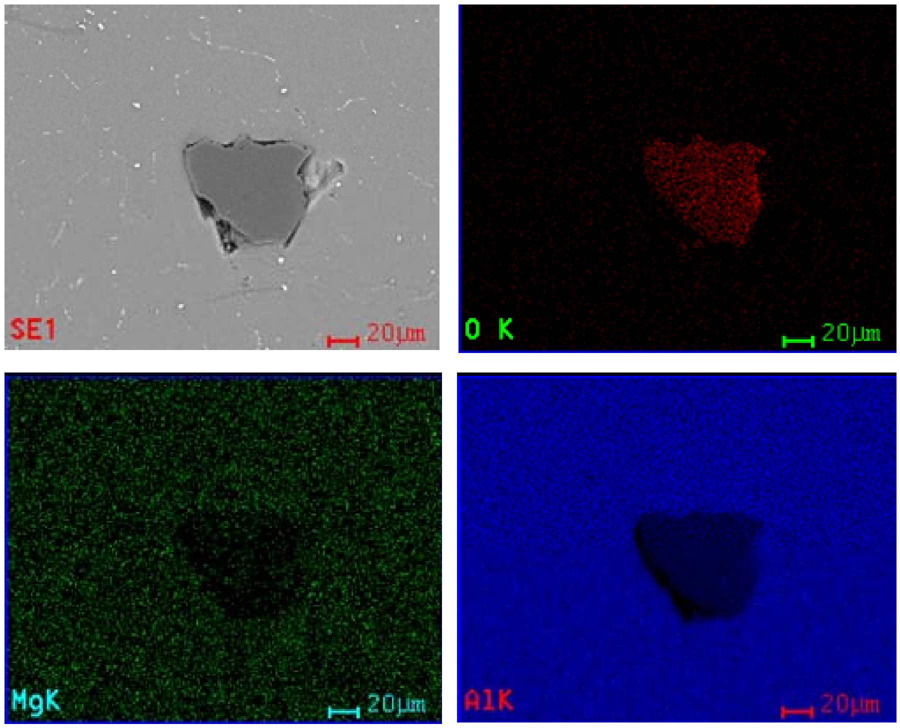

Fig. 4 X-ray element (OKα, MgKα and AlKα) distribution map in microzone of Fig. 2

X-ray images of OKα, MgKα si AlKα, confirm the presence of Al_2O_3 particle in the samples. SEM microstructure at the ceramic filter / alloy interface is given in Fig. 5.

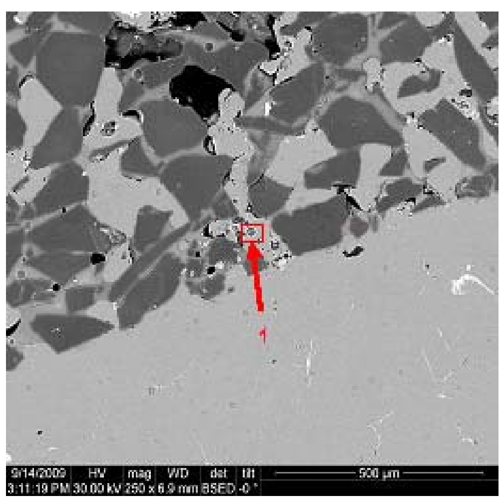

Fig 5. SEM analysis of Sample 1 at the ceramic filter/alloy interface

At the interface, in microzone 1, SEM analysis highlight agglomerations of black, white and gray particles (Fig. 6). Fig. 6 shows a classic example of spinel particles ($MgAl_2O_4$), almost black in color which are most detrimental in the aluminium alloys because of their large size and hardness. Spinel inclusions are formed from the reaction between MgO patches and aluminum oxydes:

$$MgO + Al_2O_3 = Mg\ Al_2O_4 \quad (2)$$

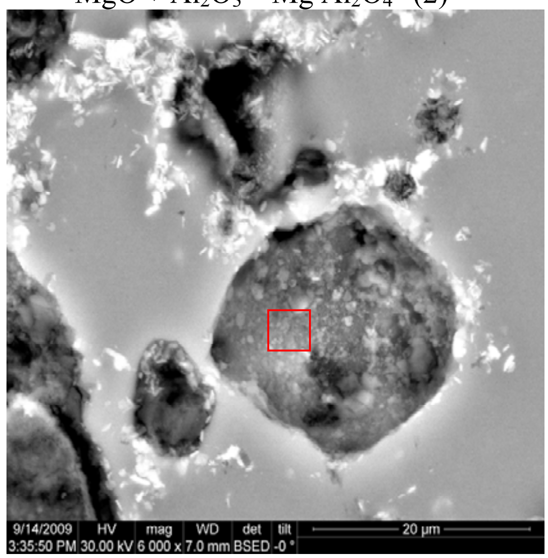

Fig. 6 SEM analysis of the microzone 1 in fig. 5

Also, agglomerations of fire white TiB_2 grain refiner particles that passed through the filter are clear observed. X-ray element distribution map for microarea of Fig. 6 is presented in Fig. 7.

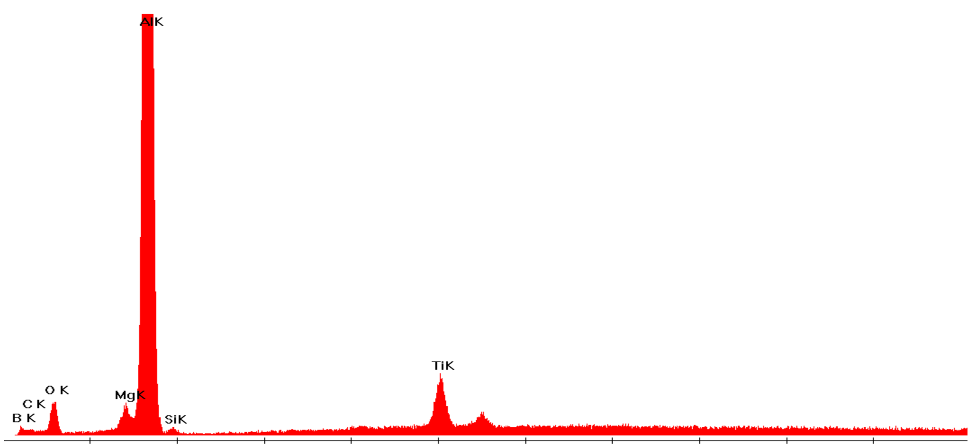

Fig.7 – EDS microanalysis of the particles included in square of Fig. 6

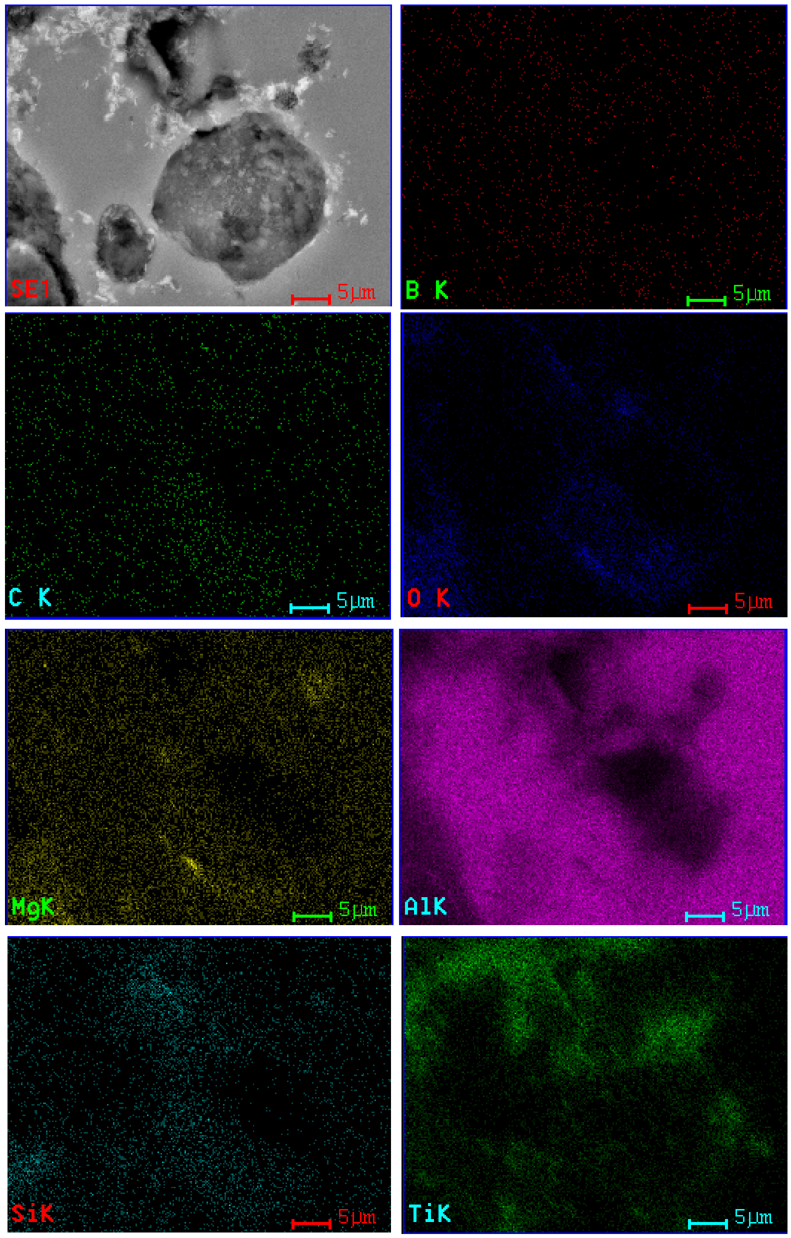

Fig.8 . X-ray element distribution map (B, C, O, Mg, Al, Si, Ti) in microzone 1 of Fig. 5

EDS microanalysis inside the square of Fig. 6 reveals a high content of Al and Ti and a low content of other elements: (B, C, O, Mg and Si).

4. Conclusions

- It was studied the nature and the contents of non-metallic inclusions in cast sleb AA5038 alloys in Wagstaff instalation at 680-690^o C.
- The analyzed samples by GRID method, taken after filtration, they have a content of non-metallic inclusions in admissible limits (0,012 mm2/kg and 0.024mm2/kg) for plastic deformation of sleb of Al-Mg-Mn alloys.
- The main determined inclusion by electron microscopy analysis (SEM, EDS, X-ray images) is Al_2O_3, which can provide from electrolysis raw material (alumina undissolved in salts bath) since it is larger (80-100μm) as used ceramic filter pores, or can appears from refractory material filter or from casting tray.
- At the ceramic filter / 5038 alloy interface, there are spherical agglomerates of magnesium and Al silicate, and particles of TiB_2 results from AlTi5B1 master alloys.
- Unlike the optical microscopy analysis, depth electron microscopy analysis can highlight more clearly the nature of non-metallic inclusions from Al-Mg-Mn alloy, creating a good premise for effective action to decrease the contamination degree of and improvement of cast sleb plasticity. [8]

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