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THE EFFECT OF TiB₂ GRANULES ON METAL QUALITY

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Abstract

TiB₂ granules were added to a fully graphitized electrolytic cell in a trial to provide a barrier coating on the carbon cathode to prolong cathode life. The consequential impact on metal cleanliness was evaluated by a detailed metallographic analysis using the PoDFA technique. Metal produced from the test cell was mixed with regular potline metal and cast into billets. Samples were taken from different locations in the process stream and also for three different types of metal charged into the furnace, namely regular potline metal, 25% of metal from test cell mixed with regular potline metal, and 50% of metal from test cell mixed with regular metal. The PoDFA analysis shows that samples containing metal from test cell had more grain refiner inclusions than regular potline metal but fewer carbide inclusions. However, there was no overall significant negative impact on the specified requirements of metal cleanliness.

Introduction

It has been shown in another paper published in these proceedings [1] that addition of TiB₂ granules was effective in reducing cathode erosion significantly. But it was required also that addition of TiB₂ should not affect hot metal quality in terms of it being inclusion free since significant deterioration would negatively affect the cast house products and consequently acceptability to the end user. To examine this aspect, the quality of hot metal tapped from the test cell was studied. Samples were taken from a control cell and from routine production for comparison. The samples were analysed using light microscopy combined with Porous Disc Filtration Apparatus (PoDFA) as well as Scanning Electron Microscope (SEM) / Energy Dispersive X-ray analyser (EDX).

Inclusions and metal cleanliness – an overview

Any foreign phase occurring in aluminium is defined as a non-metallic inclusion [2], based on the criteria that the phase already existed above the formation temperature of the first solid particle arising out of the melt. A recent thesis provides a good description of the various terms [3]. Many of these inclusions degrade the mechanical properties of the end product at aluminium foundries, extruders etc., [4,5] and also result in undesirable blemishes. In critical applications, any inclusion with diameter greater than 10 - 20 μ is considered to be deleterious although even smaller inclusions can cause problems if present in sufficient quantity. Inclusions present in fluxed and filtered aluminium metal typically range from 5 to 50 ppbv. Since this is extremely small, pre-concentration is required for metallographic study, and techniques like PoDFA are employed for this purpose [6,7,8]. Prior to the cast, from the launder, liquid metal can be sampled

and examined for presence of foreign material or inclusions by such methods.

The characteristic of metal being free of inclusions is generally referred to as “metal cleanliness”.

Classification and effect of inclusions

Different types of non-metallic inclusions and their effects on end product quality in aluminium have been described [2].

1. Oxide films classified by length and thickness films.
2. Oxide flakes consisting of magnesium and aluminium oxides.
3. Spherical oxide particles.
4. TiB₂ clusters. These are not considered very important.
5. Particles of salt inclusions soluble in water.
6. Other non-metallic inclusions such as carbide

The effect of inclusions will depend on their nature [2].

Since titanium boride is used as grain refiner in rod form, these agglomerations are considered less important. Titanium vanadium diboride (Ti,V)B₂ is said to be hard in nature and small in size but the clusters they form are tolerated at levels much higher than oxides [9]. However, in the presence of oxide films and / or liquid chloride inclusions, they can result in complex agglomerates of 20 μ to a few mm and are extremely detrimental, especially in rolled products. Magnesium oxide inclusions are said to affect downstream process only if present in large patches. Spinel inclusions are also especially harmful to the process. Particles of salt contribute to the rejection rate of aluminium sheet and bar products. Carbide inclusions can degrade mechanical properties of the final product [10].

TiB₂ inclusions / agglomerates

Since the trial involved addition of TiB₂ granules to the test cell a more detailed discussion of its effect on metal quality is relevant. TiB₂ particles when present in molten aluminium tend to form agglomerations. Since titanium boride is also used as grain refiner in casting process, this aspect is a concern for manufacturers and end users of products. Agglomerations of TiB₂ can cause end product quality problems in certain applications since TiB₂ particles are much harder than aluminium. Individually they are generally sub-micron in size, and when examined by microscope the grain refiner rods are rarely observed to be > 2 μ. The agglomerations formed can be an order of magnitude larger than the individual particles or even greater [11]. The affinity of TiB₂ particles and oxide films to agglomerate is apparent from investigation of both the microstructure of grain refiners and autopsies of tears or pinholes in foil or bright trim products. The need to minimise the level of oxide films in the casthouse has long

been recognised [12]. In many applications avoidance of TiB_2 agglomerations at the surface is important.

Description of the cast house processes at the site

The process flow sheet is presented in Figure 1.

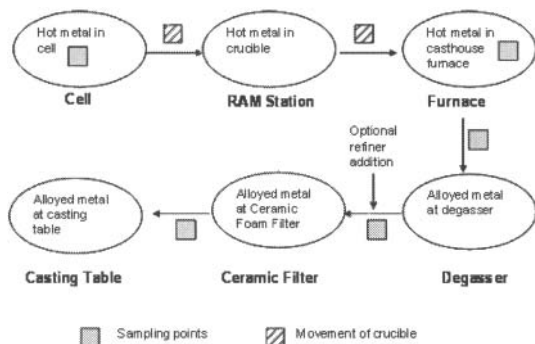


Figure 1: Cast house process flow chart

Typically, molten metal from an electrolysis cell is tapped in to a crucible of 5 -8 tonne capacity. The tapping frequency from each cell is once in 32 hours. Three to four cells are tapped to fill the crucible. Once the crucible is full, the molten metal is transported to the processing station for removing alkali metals, especially sodium by Removal of Alkali Metal (RAM) system [13,14] prior to reaching casthouse. Treated metal from several crucibles is poured in to the cast house furnace having capacity of about 50 tonnes. The molten metal is held in this furnace and mixed with alloying elements to manufacture special alloys.

Metal is then passed through degasser to remove dissolved hydrogen gas and filtered [15,16] and finally cast in to extrusion billets or other products such as foundry ingots or sows. Prior to final cast, grain refiners [17,18] are occasionally added to improve the grain structure of the finished product.

Adaptation of cast house process for experiments

It was essential to study the metal cleanliness using metal from test cell. Since the cast house furnace required 40 to 70 tonnes per batch and the cell produced only 2.5 tonnes per tap, this study was challenge. To partly circumvent this problem, production from test cell was cast into sows of 500-650 kg. About 30 tonnes of metal from the test cell was thus converted to sows for further study in casthouse. Once the treated metal was cast into sow, RAM treatment had necessarily to be skipped. RAM treatment of the ~ 2.5 tonnes tapped metal from test cell was not possible due to the design limitations of the RAM process equipment.

These sows were used in two experiments at one of the furnaces in casthouse from which molten metal was sent for extrusion billet production. Metal cleanliness standard for extrusion billets is much more demanding than for most other products and hence it was decided to use the metal from the test cell in the billet production. Melting any other cold metal during the experiments was avoided to rule out interference. Prior to the experiments, the furnace was hot cleaned. In addition, general cleaning of other refractory such as launder, degasser's trough, filter box and

casting table was also done to ensure minimisation of external contamination so that the effect of metal from test cell on billet quality could be detected easily.

In Experiment 1, 25% cold metal from test cell was mixed with 75% molten metal from other cells in same potline. All metals were charged into 40 tonnes capacity furnace and alloyed. After achieving the desired alloy composition the furnace was kept on hold for 60 minutes prior to billet casting. During the casting some inline treatments were carried out. These included grain refiner addition [19] into the casting launder before degasser, degassing metal using Alcan Compact Degasser (ACD), and finally filtering the metal through 30 ppi Ceramic Foam Filter (CFF). Control 1 was for billet production done with routinely produced hot metal from the same potline but without any metal from test cell. In Experiment 2, 50% cold metal from test cell was mixed with 50% molten metal from other cells in same potline. Control 2 was for billet production done with hot metal from same potline but without any metal from test cell.

Sampling and measurement methodology

Samples were taken from cells and at several points in the process using PoDFA unless specified otherwise (Figure 1).

Sampling from cells

For the test cell, few days after the first addition of TiB_2 , hot metal sampling from the cell was started. Samples were taken every month for a period of one year. The control cell was sampled similarly. Pot metal elemental purity was monitored for both cells as a routine measure using in house methods [20].

Sampling from crucible –after RAM

It was not possible to get 7.5 tonnes of metal exclusively from the test cell at one time. Hence 2.5 tonnes of metal from the test cell along with 5 tonnes of metal from neighbouring two cells had to be sent for RAM processing. Inclusions were measured before and after RAM treatment.

Samples from casthouse for metal cleanliness

Samples were taken from cast house furnaces while conducting Experiments 1 and 2. Furnace samples were also taken from routine production furnaces utilising hot metal from same potline for comparison. During casting, samples were taken using PoDFA at two time intervals. First set of samples was taken when cast length reached about 1.5 meter and another towards end of casting with another 1 meter left to cast. Samples were taken at outlet of furnace and CFF.

Parameters measured

Boron and titanium concentration

These concentrations were measured for metal from furnace using arc spark emission spectroscopy.

Concentration of inclusions by PoDFA

Typical operation of PoDFA has been described in literature [4]. PoDFA disc of 25 mm diameter was polished using standard procedures [21,22,23] and viewed under light microscope at different magnifications to get an overall idea of the extent of inclusions in the sample. Photographs were taken.

Characterisation of inclusion types

For every sample, in addition to total inclusions count, inclusions were also classified [24]. The inclusions were quantified by using light microscope and grid method. The counting for a particular field was done for every type of inclusion using Alcan catalogue of inclusions [9]. Identification of the inclusions was by light microscope [25] as well as SEM / EDX. Characterisation was based on the interpretation by experienced metallurgists using information such as alloy type, sampling position, metallurgy process data, inclusion colour / shape, hardness and distribution.

Results and discussion

Metal sampled directly from cells

The comparison of the results based on 12 samples is presented in Table 1. Detailed classification data for inclusions was not available for all the 19 routine production samples, but based on available data it was seen that this was similar to control cell with about 89-96 % of carbides and about 5-10 % of alumina and other inclusions with no presence of (Ti,V)B₂. After RAM treatment inclusions were reduced even further, so the levels seen in test cell did not pose any concern. Values for control cell are based on a single sample only.

Table 1: Comparison of metal cleanliness at potlines

	Unit	Test cell Average(SD)	Control cell	Routine Prodn.
Number of samples	No.	12	1	19
Total inclusion in metal	mm ² /kg	0.901 (0.513)	0.885	0.908 (0.308)
(Ti,V)B ₂	%	64 (12)	0	-
Oxide	%	2 (2)	0	-
Al ₄ C ₃	%	22 (10)	90	-
Al ₄ C ₄ B	%	4 (5)	0	-
Alumina	%	1 (1)	5	-
Others	%	7 (3)	5	-

This is further described in Figure 2. All the pinkish particles are boride inclusions and specifically (Ti,V)B₂. The grey particles are carbide inclusions. For control cell it is seen that 90 % of total inclusions are carbide inclusion but for the test cell the aluminium carbide content dropped to 22 %. It is hypothesized that the TiB₂ granules, which were added into test cell, settle down and cover the cathode in the bottom of cell and prevent aluminium carbides to be released into the bath. This suggests that the carbide transfer to bath is retarded. This is a positive sign that settled TiB₂ may be reducing carbide corrosion. And at the same time based on much higher value of (Ti,V)B₂ for test cell compared to control cell, it is postulated that TiB₂ dissolved in metal has reacted with vanadium in pot metal and produced (Ti,V)B₂. It is known that many transition metal borides including vanadium are insoluble and precipitate out when boron is added to aluminium metal[26]. The free energies of formation of VB₂ as well as TiB₂ are both negative at 970 °C.

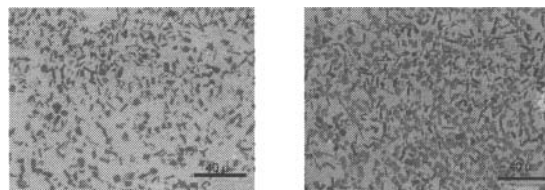


Figure 2: Carbide inclusions-control cell (left) and test cell (right)

Before and after RAM treatment

Result presented in Table 2 is for metal from one crucible based on three cells including the test cell. The total inclusion counts as well as classification according to inclusion type is shown. The reduction seen in total inclusions is in line with findings of 43 % reduction in total inclusions after RAM treatment [27].

Table 2: Metal cleanliness of crucible sample for test cell – before and after RAM

	Unit	Before RAM	After RAM
Total inclusion in metal	mm ² /kg	1.287	0.578
(Ti,V)B ₂	%	7	4
Al ₄ C ₃	%	75	53
Al ₂ O ₃	%	0	31
Alumina dispersed	G-%	13	6
Others	%	4	5

There is only 7 % (Ti,V)B₂ for crucibles which were not RAM treated but 75 % aluminium carbides are seen. Since there is practically no (Ti,V)B₂ in regular pot metal amount present in metal from test cell gets diluted since 2.5 tonnes of this gets mixed with 5 tonnes tapped from adjacent cells. Quantities of (Ti,V)B₂ work out to 0.023 and 0.090 mm²/kg in metal from test cell and diluted cell respectively and this is approximately matches with the expected figure from dilution. After RAM it is seen that aluminium carbides as well as (Ti,V)B₂ dropped and in aluminium oxides increased. It is seen that RAM system also reduced inclusions.

At Cast house

Experiment 1: cast house results - inclusions

The average measured data at the specific locations for Experiment 1 and is presented along with Control 1 in Table 3. Based on this data it was concluded that the addition of TiB₂ to test cell did not cause any significant increase in total inclusions as compared to control. The amounts observed in Experiment 1 were considered quite acceptable by cast house. The specifications for inclusions vary a great deal and are customer specific. In casthouse for the total inclusions count, international norms are 0.1-0.3 mm²/kg [28].

Experiment 2: Cast house results - inclusions

The average measured data for the same individual location for Experiment 2 is presented in Table 4 along with control sample. This experiment provided further support to concluding that the addition of TiB₂ to test cell did not cause any significant increase in total inclusions as compared to control. The differences in the results of Experiments 1 and 2 are due to other factors that affect

metal cleanliness in casthouse such as furnace cleanliness, casting parameters etc. Therefore it is concluded that using 25- 50% of metal from treated cell in one batch of about 40 tonnes will not affect the total amount of inclusions significantly as this value is within the typical range mentioned earlier. The amounts observed in Experiment 2 were considered quite acceptable by cast house. Normally, PoDFA sampling is done in cast house at furnace outlet after titanium boride grain refiner addition. For Experiment 2, in addition to this normal sampling, samples were taken before titanium boride grain refiner addition as well. This was to see the differences between borides that are coming from treated cell from those are picked up in casthouse after addition of titanium boride grain refiner (AlTi5B1). This is summarized in Table 5.

Table 3: Metal cleanliness – Experiment 1 (Average of 2 samples) & Control 1

	Unit	Furnace outlet (after AlTi5B1)		CFF outlet	
		Expt. 1	Ctrl. 1	Expt. 1	Ctrl. 1
Total inclusion	mm ² /kg	0.197	0.202	0.056	0.060
TiB ₂	%	50	37	60	61
(Ti,V)B ₂	%	13	2	10	3
Thin & thick oxide films	%	2	6	4	11
Al ₄ C ₃	%	10	14	9	9
MgO, dispersed	%	19	0	7	0
MgO, patch	%	0	25	3	4
Spinel	%	1	3	2	0
Others	%	4	13	5	10

Table 4: Metal cleanliness – Experiment 2 (Average of 2 samples) & Control 2

	Unit	Furnace outlet (after AlTi5B1)		CFF outlet	
		Expt. 2	Ctrl. 2	Expt. 2	Ctrl. 2
Total inclusion	mm ² /kg	0.537	0.559	0.295	0.263
TiB ₂	%	46	27	59	66
(Ti,V)B ₂	%	12	3	7	3
Thin & thick oxide films	%	7	9	5	6
Al ₄ C ₃	%	13	31	7	12
MgO, dispersed	%	0	0	1	1
MgO, patch	%	9	13	9	4
Spinel	%	0	6	0	0
Others	%	13	11	12	7
Graphite particles	%	0	0	0	1

Table 5: Borides before and after AlTi5B1 addition

Experiment 2 – Additional check					
	Unit	Furnace outlet (before AlTi5B1)		Furnace outlet (after AlTi5B1)	
		Sample 1	Sample 2	Sample 1	Sample 2
Total inclusion in metal	mm ² /kg	0.768	0.719	0.608	0.466
TiB ₂	%	35%	24%	50%	41%
(Ti,V)B ₂	%	21%	19%	10%	14%

The TiB₂ is generally not considered as a detrimental inclusion as it is deliberately added for grain refinement. But it was included in the total inclusion counts in order to target high standards of metal cleanliness. The oxide films were also included in the PoDFA count. The variation in the inclusions from sample to sample is due to non uniform distribution of inclusions in the bulk.

Trace elements pick up in casting furnace

It is well-known that chemical trace elements such as Ti, V, Na, etc., influence the properties of the final products at the cast house and for the end user [29]. Therefore it was needed to ensure that TiB₂ treatment in test cell should not affect metal quality. This aspect was examined for Experiments 1 and 2, compared with controls.

For each experiment, three samples were cut from the sows produced exclusively from test cell. Two samples were also taken from casting furnace after re-melting these sows. These samples were analyzed by Arc/ Spark Optical Emission Spectrometry. It is seen from average results described in Table 6 and Table 7 that B and Ti content in cast house metal are similar to the metal from other cells where no TiB₂ was added. Hence it can be concluded that there is no significant negative impact on metal chemistry by using TiB₂.

Table 6: Metal purity at cast house – Experiment 1

Element	Alloy specifications, %	Experiment 1 (average), %	Control 1 (average), %
B	-	0.0001	0
Ti	0.0030-0.0200	0.0051	0.0111
Mg	0.4700-0.5300	0.5062	0.5045
Si	0.4200-0.4800	0.4496	0.5945
Fe	0.1500-0.1900	0.1586	0.1729

Table 7: Metal purity at cast house – Experiment 2

Element	Alloy specifications, %	Experiment 2 (average), %	Control 2 (average), %
B	-	0.0001	0
Ti	0.0000-0.0200	0.0065	0.0059
Mg	0.4500-0.5100	0.4776	0.483
Si	0.4000-0.4600	0.4182	0.4267
Fe	0.1500-0.1900	0.1755	0.1795

Electrical conductivity

Electrical Conductivity of billet was measured by conductivity meter model Auto Sigma 3000 manufactured by GE Inspection Technologies [30]. There was no difference in conductivity observed between the billets containing metal from the test cell as compared to that produced with metal from regular production and the reading was 62 IACS units for both.

Grain size of homogenized billet

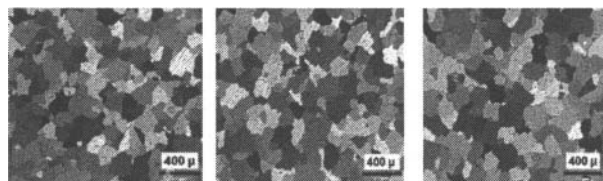
A very important property of the structure of billets is the size and shape of the grain. The morphology of grain is also important and should not be feathery or columnar in shape. Grain size of homogenized billet was measured by light microscopy and CLEMEX Image Analyser using Feret method. The main difference here was that the samples are cut from a cast billet. The results are presented in Table 8.

Table 8: Grain size of billet from Experiments 1 & 2

	Average (SD) grain size, μ			Acceptance Criteria grain size μ
	Edge	Mid	Center	
Routine production -11 samples	135(15)	137(17)	149(15)	≤ 200
Experiment 1	115	110	128	
Experiment 2	147	159	188	

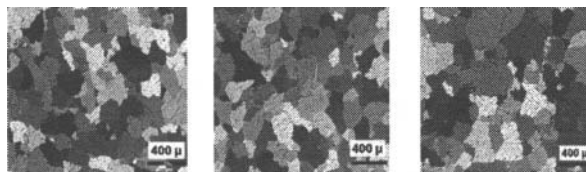
The above results were found to be within acceptance limit and similar to routine production samples for same type of alloys. The micrographs for the two experiments and for routine production are presented in Figure 3 onwards.

The grain size and the distribution were found to be similar to those from routine production. From this study it was concluded that addition of TiB_2 to the test cell did not affect the grain structure of end product.



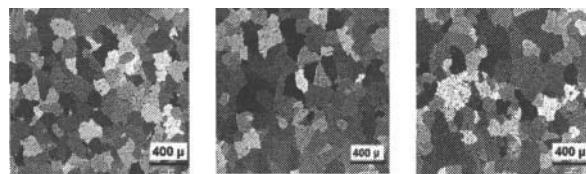
Edge 50X Mid radius 50X Centre 50X

Figure 3: Grain configuration and morphology for Experiment 1



Edge 50X Mid radius 50X Centre 50X

Figure 4: Grain configuration and morphology for Experiment 2



Edge 50X Mid radius 50X Centre 50X

Figure 5: Grain configuration and morphology for routine production

Confirmation of analytical technique

To confirm the metal cleanliness results, 11 PoDFA samples from above were sent to an accredited laboratory for inclusions count, & break-up, and metallographic analysis. Results indicated that inclusions measured (mm^2/kg) were within the range of reproducibility allowance specified by the PoDFA manual.

Conclusions

- The total amount of inclusions for pot metal from test cell remained more or less same as control cell where no TiB_2 was added. It was also within typical range of 0.3-1.5 mm^2/kg .
- Aluminium carbide was much less at 22 % for test cell than control cell where it was the main inclusion at 90 %.
- Titanium vanadium diboride (64 %) was the main type of inclusion observed in metal from test cell, which was not observed in metal from control cell at all. It is postulated that TiB_2 granules, added into test cell settled on top of the cathode and prevented further reaction of cathode with aluminium thus hindering aluminium carbide formation. Some of the TiB_2 may have reacted with vanadium in the metal to form $(Ti,V)B_2$.
- Based on the two trials in cast house it is seen that the total amount of inclusions remained more or less same irrespective of metal from test cell being used or not. Titanium diboride (about 60 %) was the main type of inclusion observed at CFF outlet for the two trials in casthouse. This is quiet normal as titanium boride is added as a grain refiner into the launder in the liquid metal at furnace outlet. It is also seen that the CFF is able to remove the inclusions quite effectively. Higher quality grades of CFF may be able to counter act higher inclusion levels

- e. As far as metal impurity levels were concerned, no negative impact was observed in casthouse based on the two trials carried out. No significant B and Ti pick up was seen in cast house metal.
- f. No impact was seen on grain size of the billets for billets produced from the two trials and hence the test did not affect this property.
- g. The conductivity data also remained similar for the metal from test cell and the control cell implying that the test did not affect the conductivity.
- h. Due to insufficient quantity, the effect of inclusions in test metal could not be evaluated by using 100 % of this material in cast house, and this would have been a more rigorous test. But on the other hand there was no RAM treatment of the metal prior to the results and the metal did not have any flux addition during processing and was processed in one of the casting stations which historically produced more non conforming product. These aspects may have counter balanced each other to some extent.
- i. Inclusions in metal are influenced by other factors such as casting station, quality of CFF used, type of degassing facility used, vibration in the metal filtration area etc., and hence the cast house can control inclusions to some extent by fine tuning the process.

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References

- 1 Part 1 of this paper, "Simplifying Protection System To Prolong Cell Life" by M.M.Al-Jallaf, M.Hyland, B.Welch, A. Al Zarouni, and A. Fahimi being published in TMS 2010.
- 2 J.Langerweger, Light Metals 1981, 688- 705.
- 3 N.Habibi, Ph.D Thesis, Université du Québec à Chicoutimi (UQAC), Canada, August 2002, 15-23.
- 4 D.Doutre, B.Gariepy, J.P.Martin and G. Dube, Light Metals 1985, 1179-1189.
- 5 "The Influence and Control of Porosity and Inclusions in Aluminium Castings", Downloaded from ASM website on 15-11-09.
http://www.asminternational.org/pdf/spotlights/5114alum_castc5.pdf
- 6 L.Liu and F.H.Samuel, Journal Of Materials Science, 32 (1997), 5901 – 5925.
- 7 L.Liu and F.H.Samuel, Journal Of Materials Science 32 (1997) 5927- 5944.
- 8 "Determination of PoDFA Inclusion Concentrates", Alcan Report AR-90/0032, Dec 1990 (Confidential).
- 9 M.Ryvola, "Catalogue of Inclusions in Aluminium and its Alloys", Alcan International Limited, Revised Version – November 1991, 78 pages.
- 10 J.Rødseth, B.Rasch, O.Lund and J.Thonstad, Light Metals 2002, 883-887.
- 11 P. Cooper and A. Barber, "Review of the Latest Developments and Best use of Grain Refiners", 2nd International Melt Quality Workshop, Prague, Czech Republic, 16-17th October 2003, 10 pages.
- 12 Q.G.Wang, D.Apelian and D.A.Lados, J. Light Metals, 2001, 73-84.
- 13 B. Rasch, E.Myrbostad and K.Haf, Light Metals 1998, 851-854.
- 14 "Hycast RAM System User Manual", Hycast AS, Hydro Aluminium, Revision 0, 2007.
- 15 C.E.Eckert, R.E.Miller, O.Apelian and R.Mutharasan, Light Metals 1984, 1281-1304.
- 16 Y.Ohno, Journal of Japan Institute of Light Metals, No.51, 2001, 134-137. off print from Pyrotek company website, 1-10-09 (English translation of Japanese paper).
- 17 M.Bryant and P.Fisher, "Grain Refining And The Aluminium Industry - Past. Present And Future", Proc. 3rd Austr. Alum. Conf., editor Nilamani, TMS 1993, 281-291.
- 18 A..M.Detomi, A.J.Messias, S.Majer and P.S.Cooper, Light Metals 2001, 919-926.
- 19 P.Cooper and A.Barber, 2nd International Melt Quality Workshop, Prague, Czech Republic, 16-17th October 2003, 10 pages.
- 20 DUBAL Internal procedure PR/TSK.14.
- 21 C.Kammer, "Aluminium Handbook", vol. 1, 1st. ed. , Aluminium Verlag GmbH,1999, 511-520.
- 22 M.Warmuzek, "Metallographic Techniques for Aluminum and Its Alloys, Metallography and Microstructures, vol. 9, ASM Handbook, ASM International, 2004, p. 711-751.
- 23 J.E.Hatch, "Aluminium Properties and Physical Metallurgy, Chapter 3 : Microstructure Of Alloys", American Society for Metals, 1984, 58-104.
- 24 "PoDFA Inclusion Catalog", ARVIDA Research and Development Centre, ALCAN International Limited, 07-10-1997, 27-42 & 47.
- 25 "Metallography and Microstructures", Metals Handbook, Ninth Edition, vol.9, American Society for Metals, United States of America, 1985.
- 26 S.Karabay, Materials & Design, vol. 29, 2008, 1364-1375.
- 27 Internal plant data at DUBAL.
- 28 M.V.Canullo and R.A.Laje, "Metallurgical Quality of Aluar Billets , Ten Years of Increasing Quality", ET08 -9th International Aluminium Extrusion Conference, May 2008, 15 pages.
- 29 C.J.Simensen, "Sources of Impurities in Aluminium Melts and Their Control", Aluminium Melt Refining and Alloying – Theory and Practice, Melbourne, July 10-12, 1989, C1 –C19.
- 30 "Auto Sigma 30000 ;Technical and Operations Reference Manual", GE Inspection Technologies, LP, USA, Issue 04, 05/2005.