



(11) **EP 2 446 065 B2**

(12) **NEW EUROPEAN PATENT SPECIFICATION**
After opposition procedure

(45) Date of publication and mention
of the opposition decision:
21.02.2024 Bulletin 2024/08

(45) Mention of the grant of the patent:
16.12.2020 Bulletin 2020/51

(21) Application number: **10785623.9**

(22) Date of filing: **08.06.2010**

(51) International Patent Classification (IPC):
C22B 21/06 ^(2006.01) **C22B 9/10** ^(2006.01)

(52) Cooperative Patent Classification (CPC):
C22B 9/10; C22B 21/06; C22B 21/062

(86) International application number:
PCT/CA2010/000866

(87) International publication number:
WO 2010/142025 (16.12.2010 Gazette 2010/50)

(54) **USE OF A BINARY SALT FLUX OF NaCl AND MgCl₂ FOR THE PURIFICATION OF ALUMINUM OR ALUMINUM ALLOYS, AND METHOD THEREOF**

VERWENDUNG EINES BINÄREN NACL- UND MGCL₂-SALZFLUSSES ZUR REINIGUNG VON ALUMINIUM ODER ALUMINIUMLEGIERUNGEN SOWIE VERFAHREN DAFÜR

UTILISATION D'UN FLUX SALIN BINAIRE DE NaCl ET DE MgCl₂ POUR LA PURIFICATION D'ALUMINIUM OU D'ALLIAGES D'ALUMINIUM ET PROCÉDÉ CORRESPONDANT

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO SE SI SK SM TR

(30) Priority: **08.06.2009 CA 2668473**

(43) Date of publication of application:
02.05.2012 Bulletin 2012/18

(73) Proprietor: **Pyrotek, Inc.**
Spokane, WA 99206 (US)

(72) Inventors:
• **TREMBLAY, Sylvain**
Chicoutimi
Québec G7G 5C8 (CA)
• **DESROSIERS, Luc**
Sherbrooke
Québec J1N 2K2 (CA)

• **LEVESQUE, Daniel**
Sherbrooke
Québec J1N 4K2 (CA)

(74) Representative: **Cabinet Nony**
11 rue Saint-Georges
75009 Paris (FR)

(56) References cited:
WO-A1-2005/049875 US-A- 1 476 192
US-A- 4 138 245 US-A- 4 138 245
US-A- 4 790 873 US-A- 5 427 602

• **COX A ET AL: "Separation of Mg and Mn from beverage can scrap using a recessed-channel", JOURNAL OF THE ELECTROCHEMICAL SOCIETY, ECS, vol. 150, no. 12, 1 December 2003 (2003-12-01), pages D200-D208, XP009167862, ISSN: 0013-4651, DOI: 10.1149/1.1623768**

EP 2 446 065 B2

Description**Field of the invention**

[0001] The invention relates to the use of a binary salt flux comprising NaCl and $MgCl_2$ for the purification of a metal selected from the group consisting of aluminum alloys, for the removal of alkali and alkaline-earth metals. The invention also relates to a method for the purification of said metal with said binary salt flux.

Prior art

[0002] The use of fluxes is well known in the field of metallurgy and these fluxes fulfill various functions.

[0003] Fluxes can be used to form a protecting layer at the surface of an alloy to prevent oxidation. When fluxes contain chemical active agents, they can be used to clean furnace walls by softening accumulated layers of corundum. Some exothermic fluxes are also used for cleaning dross and removing aluminum trapped in oxide layers.

[0004] Fluxes that are based on alkali chlorides and alkaline-earth chlorides are also used for the refining of alloys. Those skilled in the art generally define refining as the removal of alkali and alkaline-earth metals, non metallic inclusions and hydrogen from the alloys.

[0005] Sodium and calcium are always present as impurities in aluminum obtained from the Hall-Heroult process. Lithium fluoride is often added to the electrolytic bath to improve the efficiency of cells. However, a small amount in the metallic state is found dissolved in the aluminum. These impurities entail quality issues. For example, in an alloy containing magnesium, the presence of sodium may interfere during the hot rolling processes. The presence of sodium in aluminum and silicon alloys neutralize the effect of phosphorus used for the refining of grains. For the above-mentioned reasons, the use of fluxes containing sodium is not recommended for aluminum and its alloys, more particularly for aluminum alloys comprising a magnesium content higher than 3 % by weight or a silicon content higher than 10 % by weight.

[0006] Also, the presence of hydrogen in too high concentration may lead to a too high porosity of the aluminum during its solidification. During the recycling of aluminum, the presence of non metallic inclusions is important.

[0007] Reactional kinetics for the withdrawal of calcium and sodium in an aluminum alloy have been well studied. Naturally, in these alloys, both impurities disappear according to a kinetic of order 1 for small concentrations and order 0 for high concentrations. Because of its high vapor tension, sodium oxidizes itself more rapidly than calcium, that is why calcium is used during cleaning tests. The addition of fluxes involves an increase of reactional constants and thereby a faster reduction of the content in impurities. Mixing also has a non negligible effect on the reduction of impurities. Mixing accelerates the withdrawal of impurities by increasing the contact between impurities and the salt flux.

[0008] $MgCl_2$ is one of the chemical active agents used for the withdrawal of impurities in alloys. Its concentration has a direct effect on the kinetic of withdrawal of calcium and sodium. Its melting point is $714^\circ C$, but in common fluxes, it is mixed with other salts to obtain a melting point between 400 and $550^\circ C$. However, $MgCl_2$ is hygroscopic and can not be exposed for a long period of time to the surrounding air. Fluxes obtained by fusion of salts comprising magnesium chloride have hygroscopic properties. Consequently, the packaging is an important factor in limiting the absorption of humidity during the manufacturing of such fluxes.

[0009] There are examples of fluxes that are based on magnesium chloride. US patent no. 1,377,374 relates to the use of a flux having an equimolar composition of sodium chloride and magnesium chloride for the production of manganese or magnesium alloys. US patent no. 1,754,788 relates to the use of this same flux in a process for the cleaning of magnesium. US patent no. 1,519,128 relates to the addition of calcium chloride to this composition and US patent no. 2,262,105 relates to the addition of potassium chloride and magnesium oxide in addition to the calcium chloride. US patent no. 5,405,427 mentions a flux based on sodium chloride, magnesium chloride, potassium chloride and carbon for the treatment of metal.

[0010] The article entitled "Salt Fluxes for Alkali and Alkali and Alkaline Earth Element Removal from Molten Aluminum" by David H. DeYoung shows the use of a ternary salt based on magnesium chloride, sodium chloride and potassium chloride for the removal of sodium, calcium and lithium from aluminum alloys. However, the article entitled "The Treatment of Liquid Aluminum-Silicon Alloys" by Gruzleski et al., pp. 204-205 indicates that it is important to those skilled in the art, not to use fluxes containing sodium salts. Therefore, even if a ternary flux salts having low content in sodium salts may be tolerated, those skilled in the art are expressly invited to avoid using sodium salts.

[0011] Initially, the refining of aluminum was carried out by bubbling of chlorine and argon in the liquid metal. However, this created environmental problems due to emissions of chlorine, chlorhydric acid and particles in suspension. The use of salt fluxes was later adopted as a more ecologically-friendly solution.

[0012] The refining fluxes are usually composed of alkali chlorides or alkaline-earth chlorides, which are mixed to obtain melting points that are lower than the operating temperature of alloys -- the melting point of pure compounds being usually quite high.

[0013] Several methods can be used to incorporate salt fluxes in an alloy. US patent no. 4,099,965 relates to a method

where a flux of KCl and MgCl_2 is added in solid form in the bottom of a preheated container before the addition of aluminum. More currently, fluxes are added by an inert gas in a pipe under the surface of the metal (lance fluxing). Recently, a method was developed where a hollow shaft brings the salt flux in the alloy with a gas carrier, and the salt flux is dispersed by an agitator (rotary flux injection). This method reduces the amount of salt flux required for carrying out the purification while increasing the dispersion of this salt flux in the alloy. Following the addition of a salt flux to the metal, impurities and salts float on the surface of the liquid metal and can be easily removed.

[0014] It is also known from US5,427,602 a method of treating molten metal containing solid particles suspended therein so as to reduce the number of said solid particles by passing the molten metal through porous media so construed and arranged such that the movement of the molten metal therethrough renders the suspended particles gravity separable, said separation being preceded by a treatment of said molten metal with a material, said treatment creating liquid salt particles in said molten metal, said liquid salt particles being suspended in said molten metal and said material being potentially a material is a chlorinaceous material such as magnesium chloride, lithium chloride, potassium chloride, sodium chloride and combinations thereof.

[0015] It is also known from the article Antony Cox "Separation of Mg and Mn from Beverage Can Scrap using a Recessed-Channel Cell" Journal of the Electrochemical Society, 150 (12) D200-D208 (2003) a separation process using a Recessed-Channel Cell.

[0016] Advantageously, the use of solid compounds obtained by melting of salts controls the granulometry. Particles may be used in batch processes or in continuous processes.

[0017] However, costs related to salt fluxes such as binary mixtures of magnesium chloride and potassium chloride, are high. Furthermore, the use of salt fluxes having a substantial content in sodium chloride is not recommended by those skilled in the art due to perceived negative effects of sodium content in the resulting aluminum or aluminum alloys. In fact, when sodium chloride is present in fluxes for the purification of aluminum or aluminum alloys, those skilled in the art currently will avoid or limit the use of sodium chloride. More particularly, in the case of certain kinds of alloys such as, for example, aluminum alloys having silicon content higher than 10% by weight and more particularly aluminum alloys having magnesium content higher than 3% by weight, those skilled in the art currently recommend not using sodium chloride in salt flux.

[0018] During Applicant's search for a more effective solution to the purification problem, it was surprisingly noted that contrary to current apprehensions and beliefs of those skilled in the art, it is possible in a salt flux containing MgCl_2 , to replace expensive KCl by inexpensive NaCl. Consequently, the present invention according to the claims offers an economical solution for the treatment of aluminum alloys with an efficiency of purification that is equivalent to methods presently used. Indeed, contrary to apprehensions of those skilled in the art, there is no significant amount of sodium in the resulting aluminum alloys when using the inventive purification method described herein.

[0019] Embodiments of the present invention show the following advantages:

- Economical advantages
 - Lower production costs because the melting point of the flux is lower.
 - Lower costs of raw material.
- Efficiency equivalent to the purification methods using an existing well known salt flux sold under the trademark Promag (40 wt % KCl - 60 wt % MgCl_2).
- Economical alternative to existing product sold under the trademark Promag without creating any significant accumulation of sodium within aluminum alloys.

Summary of the Invention

[0020] The present text describes to the use, which is not encompassed by the wording of the claims, of a salt flux for the purification of a metal selected from the group consisting of aluminum alloys, said metal being in liquid phase and said salt flux being a binary mixture of NaCl and MgCl_2 .

[0021] This text also describes the use, which is not encompassed by the wording of the claims, of a salt flux for the purification of a metal selected from the group consisting of aluminum alloys, said metal being in a liquid phase, wherein the salt flux is in the form of particles obtained by grinding a fused salt of NaCl and MgCl_2 , said binary mixture comprising from 40 to 50 % by weight of NaCl and from 50 to 60 % by weight of MgCl_2 .

[0022] The invention relates to a method according to claim 1.

[0023] The invention pertains to a method as defined hereinabove, wherein the salt flux: consists of particles resulting from the grinding of a fused salt of NaCl and MgCl_2 in solid state.

[0024] The invention relates to a method as defined in any one of the above-mentioned embodiments, wherein the binary mixture comprises: a) from 40 to 50% by weight of NaCl; and b) from 50 to 60% by weight of MgCl₂. More particularly, this binary mixture comprises 45% by weight of NaCl and 55% by weight of MgCl₂ to form an eutectic mixture having a melting point of about 439°C.

[0025] The binary mixture may comprise:

a) 45 % by weight of NaCl; and

b) 55 % by weight of MgCl₂;

to form an eutectic binary mixture having a melting point of 439°C.

[0026] Another embodiment of the invention relates to a method as defined in any one of the above-mentioned embodiments, wherein when the salt flux is in the form of particles, those particles have an average particle size between 100 μm and 3.35 mm. Preferably, said particles may have a particle size between 0.85 mm and 3.15 mm or between 100 μm and 1 mm. The present text describes such a use.

[0027] Another embodiment of the invention relates to a method as defined in any one of the above-mentioned embodiments, wherein the particles are contacted with the liquid metal by injection with a gas injection equipment. A non limiting example of a gas injection equipment may consist of a rotary injector known under the tradename SNIF PHD-50 commercialized by the Applicant. The present text discloses such a use.

[0028] Another embodiment of the invention relates to a method as defined in any one of the above-mentioned embodiments, wherein the metal is an aluminum alloy having a magnesium content higher than 3% by weight. The present text describes such a use.

[0029] Another embodiment of the invention relates to a method as defined in any one of the above-mentioned embodiments, wherein the metal is an aluminum alloy having a silicon content higher than 10% by weight. The present text discloses such a use.

[0030] The present invention will be better understood with reference to the following drawings:

Figure 1: a phase diagram of a fused salt KCl/NaCl/MgCl₂;

Figure 2: a phase diagram of a fused salt KCl/MgCl₂;

Figure 3: a phase diagram of a fused salt NaCl/MgCl₂; and

Figure 4: a comparative graphic concerning examples 5 to 8.

Phase diagrams of Figures 1 to 3 were extracted from factsage web site (<http://factsage.com>).

DESCRIPTION OF EMBODIMENTS OF THE INVENTION

[0031] In the following examples, the Applicant has noted, contrary to apprehensions of those skilled in the art, that formulations of salt fluxes comprising a binary mixture of NaCl and MgCl₂ do not involve an increase of the concentration of metallic sodium in an aluminum alloy having magnesium content. A non limiting example of such an aluminum alloy may consist of an aluminum alloy having a magnesium content of 5 % by weight. Consequently, it appears that there is no counter-indication of using a binary salt flux comprising NaCl and MgCl₂ for cleaning aluminum, especially in the case of an aluminum alloy with high magnesium content.

[0032] Formulations based on NaCl and MgCl₂ proposed according to the present invention, show melting points that are lower than those of salt flux compositions sold by the Applicant under the trademark Promag (40 wt % KCl, 60 wt % MgCl₂), for equivalent amounts of MgCl₂ which is the chemically active agent for the withdrawal of impurities. The lowering of melting points represents a lowering of energy costs when melting the solid salt flux.

[0033] Two fused salts have been evaluated in examples hereinafter, that is (on a weight basis) the following binary system salt flux and ternary system salt flux (prior art):

- 45 % NaCl and 55 % MgCl₂ with a melting point of 439°C; and
- 20 % NaCl, 20 % KCl and 60% MgCl₂ with a melting point of 396°C.

[0034] Also, example 1 illustrates an unexpected effect with regard to the sodium concentration in an aluminum alloy when NaCl is added in a liquid aluminum alloy, that is, no increase of the sodium content in the alloy obtained.

[0035] Preparation of each salt flux was made by mixing the salts in an anhydrous solid phase in an appropriate oven. Then, by increasing the temperature of the oven, a fused compound in liquid form was obtained. The liquid was then cooled down quickly, grinded and sifted to obtain a granulometry that was appropriate for the selected method. In example 7 hereinafter, the salt flux was made only by mixing the salts in an anhydrous solid phase.

[0036] Salt fluxes have shown an optimal efficiency for the withdrawal of Ca, Na and Li when used with a rotary injector such as a SNIF PHD-50 (tradename) commercialized by the Applicant (Pyrotek). Of course, other methods of addition well known to those skilled in the art and already mentioned for use in connection with prior art purification methods can be used to carry out the purification. The concentrations of salt fluxes required to carry out the purification may vary depending on the selected method.

Example 1 (out of the invention)

[0037] In a crucible made of graphite, one hundred grams (100 g) of NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL, were agitated in 1.5 kg of a liquid AA110 aluminum (sold under the trademark Alcan) in which 5 wt % of solid magnesium were added. The crucible was maintained at 850°C during the whole test. Samples were taken every day during 7 days. According to these daily analyses, the sodium content of the resulting aluminum alloy was at a minimal level of 2 ppm during the whole test, showing that, contrary to apprehensions of those skilled in the art, an addition of NaCl does not involve an absorption of sodium in an aluminum alloy with high magnesium content.

Example 2

[0038] In a graphite crucible, fifteen grams (15 g) of a salt flux consisting of a binary mixture of 45 wt % NaCl and 55 wt % MgCl₂ were agitated in 1.5 kg of a liquid AA1100 aluminum alloy (sold under the trademark Alcan) in which 5 wt % of magnesium were added. The crucible was maintained at 720°C during 90 minutes and samples were taken every 30 minutes. The sodium level in the crucible was maintained at a minimal level of 3 ppm during the whole experiment, showing that an addition of a flux comprising NaCl does not involve an absorption of sodium in an aluminum alloy with high magnesium content. The salt flux was prepared from NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL and MgCl₂ in flake form and sold under the trademark SKYLINE.

Example 3 (prior art)

[0039] In a graphite crucible, fifteen grams (15 g) of a salt flux consisting of a ternary mixture of 20 wt % NaCl, 20 wt % KCl and 60 wt % MgCl₂ were agitated and added in 1.5 kg of a liquid AA1100 aluminum alloy (sold under the trademark Alcan) in which 5 wt % of magnesium were added. The crucible was maintained at 720°C during 90 minutes and samples were taken every 30 minutes. The sodium level in the crucible was maintained at a minimal level of 3 ppm during the whole experiment, showing that an addition of a ternary flux comprising a small amount of NaCl does not involve an absorption of sodium in an aluminum alloy with high magnesium content. The salt flux was prepared from NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL, KCl in powder form and sold under the trademark IMC KALIUM and MgCl₂ in flake form and sold under the trademark SKYLINE.

Example 4:

[0040] About seventy-five kilos (75kg) of A356 alloy were melted and maintained in a liquid state at 700°C in a crucible made of silicon carbide. Then, 535g of an aluminum alloy containing 10 % by weight of calcium were added to the liquid A356 alloy while mixing it with an agitator having straight blades. Then the resulting aluminum alloy contained in the crucible was left without agitation for 5 hours. During this time, the calcium content of the resulting aluminum alloy was reduced from 350 ppm to 150 ppm. Then, three hundred and sixty grams (360g) of a salt flux made of 45 wt % NaCl and 55 wt % MgCl₂ were added to the resulting alloy while agitating it in order to further purify it. The salt flux was prepared from NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL and MgCl₂ in flake form and sold under the trademark SKYLINE.

[0041] Analyses made on the purified aluminum alloy have shown a reduction of the Ca content from 150 ppm to 70 ppm, that is a reduction of 53 %, immediately after the addition of the salt flux, and this Ca content drops to 25 ppm 3 hours after the addition. Also, analyses have shown that the sodium content was in the order of 2 ppm.

Example 5

[0042] Fifty grams of a flux were prepared in a small alumina crucible by mixing 22.5 grams of NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL, and 27.5 grams of MgCl₂ in flake form and sold under the trademark

SKYLINE. The mixture was subjected to a temperature of 550°C during 45 minutes. The liquid mixture obtained was then poured into an enamelled-coated bowl for quick solidification. The salt flux obtained was then grinded with in a mortar and sifted. The fraction having a particle size lower than 3150 microns and higher than 105 microns was recovered.

[0043] Two kg of AA1100 aluminum alloy (sold under the trademark Alcan) were melted and kept in liquid state at 700°C in a graphite crucible. To this alloy, 2 grams of an aluminum alloy consisting of 90 wt % of aluminum and 10 wt % of calcium (sold under the trademark KB Alloys) were added in a vortex formed with an agitator in the liquid metal, said agitator having straight blades. The agitation was maintained during 2 minutes. A sample of the metal was taken for analysis. Two grams of the flux formed hereinabove were added to the liquid aluminum alloy doped with calcium while agitating for 2 minutes. Samples were taken immediately after the end of the agitation as well as 30, 60 and 90 minutes later.

[0044] Analyses of samples have shown a reduction of the Ca level from 115 ppm to 3 ppm after the addition of the salt flux. Thirty minutes later, the calcium level was under 2 ppm. No increase in the sodium content was noted during the test. The level of sodium in the alloy was in the order of 2 ppm.

Example 6 (prior art)

[0045] Fifty grams of a salt flux were prepared in a small alumina crucible by mixing 10 grams of NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL, 10 grams of KCl in powder form and sold under the trademark IMC KALIUM, and 30 grams of MgCl₂ in flake form and sold under the trademark SKYLINE. The mixture was subjected to a temperature of 550°C during 45 minutes. The liquid mixture obtained was then poured into an enamelled-coated bowl for quick solidification. The salt flux obtained was then grinded in a mortar and sifted. The fraction having a particle size lower than 3150 microns and higher than 105 microns was recovered.

[0046] Two kg of AA1100 aluminum alloy (sold under the trademark Alcan) were melted and kept in liquid state at 700°C in a graphite crucible. To this alloy, 2 grams of an aluminum alloy consisting of 90 wt % of aluminum and 10 wt % of calcium (sold under the trademark KB Alloys) were added in a vortex formed with an agitator in the liquid metal, said agitator having straight blades. The agitation was maintained during 2 minutes. A sample of the metal was taken for analysis. Two grams of the salt flux formed hereinabove were added to the liquid aluminum alloy doped with calcium while agitating for 2 minutes. The agitation was stopped and samples were taken immediately after the end of the agitation as well as 30, 60 and 90 minutes later.

[0047] The analysis of samples shows a reduction of the Ca level from 108 ppm to 7 ppm after the addition of the salt flux. Thirty minutes later, the calcium level was at 2 ppm and after 60 minutes the calcium level was under 1 ppm. No increase in sodium content was noted during the test. The sodium level was in the order of 2 ppm. This example shows that a ternary flux having a low content in NaCl does not increase the level of sodium in the alloy.

Exemple 7 (out of the invention)

[0048] Fifty grams of a salt flux were prepared only by mixing 22.5 grams of NaCl in powder form and sold under the trademark SIFTO INDUSTRIAL with a granulometry 95% lower than 840 microns and 95 % higher than 300 microns, and 27.5 grams of MgCl₂ in flake form and sold under the trademark SKYLINE with a granulometry 90 % lower than 4.7 mm and 85 % higher to 1 mm.

[0049] Two kg of AA1100 aluminum alloy (sold under the trademark Alcan) were melted and kept in liquid phase at 700°C in a graphite crucible. To this alloy, 2 grams of an aluminum alloy consisting of 90 wt % of aluminum and 10 wt % of calcium (sold under the trademark KB Alloys) were added in a vortex formed with an agitator in the liquid metal, said agitator having straight blades. The agitation was maintained during 2 minutes. A sample of the metal was taken for analysis. Two grams of the salt flux formed hereinabove were added to the liquid aluminum alloy doped with calcium while agitating for 2 minutes. Agitation was stopped and samples were taken immediately after the end of the agitation as well as 30, 60 and 90 minutes later.

[0050] Analyses of samples have shown a reduction of the Ca level from 77 ppm to 2 ppm after the addition of the salt flux. Thirty minutes later, the calcium level was under 1 ppm. No increase in the sodium content was noted during the test. The sodium level was in the order of 2 ppm.

Example 8

[0051] Two kg of AA1100 aluminum alloy (sold under the trademark Alcan) were melted and kept in liquid state at 700°C in a graphite crucible. To this alloy, 2 grams of an aluminum alloy consisting of 90 wt % of aluminum and 10 wt % of calcium (sold under the trademark KB Alloys) were added in a vortex formed with an agitator in the liquid metal, said agitator having straight blades. Stirring was maintained during 2 minutes. A sample of the metal was taken for analysis. Two grams of the PROMAG SI (trademark) formed of 40 wt % KCl and 60 wt % MgCl₂, with a granulometry

99% lower than 3150 microns and 95 % higher than 850 microns, were added to the alloy doped with calcium while agitating for 2 minutes. The agitation was stopped and samples were later taken immediately after the end of the agitation as well as 30, 60 and 90 minutes later.

[0052] Analyses of samples have shown a reduction of the Ca level from 75 ppm to 7 ppm after the addition of the salt flux. Thirty minutes later the calcium level was under 5 ppm (see Figure 4). These analyses show that binary fluxes of NaCl and MgCl₂ are more efficient than a ternary flux of NaCl, KCl and MgCl₂ or binary flux, KCl and MgCl₂.

	Ex. 5 NaCl- MgCl ₂ (45-55) Fused	Ex. 6 NaCl-KCl-MgCl ₂ (20-20-60) Fused	Ex. 7 NaCl-MgCl ₂ (45-55) Mix	Ex. 8 Promag SI* (KCl - MgCl ₂) (40-60)	Reference (no flux)
Time					
After Ca	115	108	77	75	77
After salt	3	7	2	7	-
30 min	<1	2	<1	<5	58
60 min	<1	<1	<1	<5	-
90 min	<1	<1	<1	<5	-
120min	-	-	-	-	39

* trademark

[0053] The present invention has been described with respect to its preferred embodiments. The description and the drawings are only intended to aid to the understanding of the invention and are not intended to limit its scope. It will be clear to those skilled in the art that numerous variations and modifications can be made to the implementation of the invention without being outside the scope of the invention. Such variations and modifications are covered by the present invention. The invention will be now described in the following claims:

Claims

1. A method for the purification of a metal with a binary salt flux, the metal being selected from the group consisting of aluminum alloys, for the removal of alkali and alkaline-earth metals, wherein said method comprises:

• heating the metal to a liquid phase; and

contacting the liquid metal with the salt flux consisting of a binary mixture of NaCl and MgCl₂, wherein said salt flux consists of particles resulting from the grinding of a fused salt of NaCl and MgCl₂ in solid state said binary mixture comprising from 40 to 50 % by weight of NaCl and from 50 to 60 % by weight of MgCl₂.

2. The method according to claim 1, wherein said particles have an average particle size comprised between 100 μm and 3.35 mm, in particular between 0.85 mm and 3.15 mm and more particularly between 100 μm and 1 mm.

3. The method according to anyone of claims 1 or 2, wherein said particles are contacted with the liquid metal by injection with a gas injection equipment.

4. The method according to any one of claims 1 to 3, wherein the metal is an aluminum alloy having a magnesium content higher than 3 % by weight.

5. The method according to any one of claims 1 to 4, wherein the metal is an aluminum alloy having a silicon content higher than 10 % by weight.

Patentansprüche

1. Verfahren zur Aufreinigung eines Metalls mit einem binären Flussmittel auf Basis von Salz, wobei das Metall aus der Gruppe bestehend aus Aluminiumlegierungen ausgewählt wird, zur Entfernung von Alkali- und Erdalkalimetallen, wobei man bei dem Verfahren:

- das Metall bis in den flüssigen Zustand erhitzt und
- das flüssige Metall mit dem Flussmittel auf Basis von Salz, das aus einer binären Mischung von NaCl und MgCl₂ besteht, in Kontakt bringt, wobei das Flussmittel auf Basis von Salz aus Teilchen besteht, die aus dem Mahlen einer Salzschnmelze von NaCl und MgCl₂ im festen Aggregatzustand erhalten wird, wobei die binäre Mischung 40 bis 50 Gewichts-% NaCl und 50 bis 60 Gewichts-% MgCl₂ umfasst.

2. Verfahren nach Anspruch 1, wobei die Teilchen eine durchschnittliche Teilchengröße aufweisen, die zwischen 100 µm und 3,35 mm, insbesondere zwischen 0,85 mm und 3,15 mm und ganz besonders zwischen 100 µm und 1 mm liegt.
3. Verfahren nach Anspruch 1 oder 2, wobei man die Teilchen mit dem flüssigen Metall durch Eindüsung mit einer Gaseindüsungsvorrichtung in Kontakt bringt.
4. Verfahren nach einem der Ansprüche 1 bis 3, wobei es sich bei dem Metall um eine Aluminiumlegierung mit einem Magnesiumgehalt von mehr als 3 Gewichts-% handelt.
5. Verfahren nach einem der Ansprüche 1 bis 4, wobei es sich bei dem Metall um eine Aluminiumlegierung mit einem Siliciumgehalt von mehr als 10 Gewichts-% handelt.

Revendications

1. Procédé pour la purification d'un métal avec un flux salin binaire, le métal étant choisi dans le groupe constitué par des alliages d'aluminium, pour l'élimination de métaux alcalins et alcalino-terreux, ledit procédé comprenant :
 - le chauffage du métal en une phase liquide ; et
 - la mise en contact du métal liquide avec le flux salin constitué d'un mélange binaire de NaCl et MgCl₂, dans lequel ledit flux salin est constitué de particules résultant du broyage d'un sel fondu de NaCl et de MgCl₂ à l'état solide, ledit mélange binaire comprenant de 40 à 50 % en poids de NaCl et de 50 à 60 % en poids de MgCl₂.
2. Procédé selon la revendication 1, dans lequel lesdites particules ont une taille moyenne de particule comprise entre 100 µm et 3,35 mm, en particulier entre 0,85 mm et 3,15 mm et plus particulièrement entre 100 µm et 1 mm.
3. Procédé selon l'une quelconque des revendications 1 ou 2, dans lequel lesdites particules sont mises en contact avec le métal liquide par injection avec un appareillage d'injection de gaz.
4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel le métal est un alliage d'aluminium ayant une teneur en magnésium supérieure à 3 % en poids.
5. Procédé selon l'une quelconque des revendications 1 à 4, dans lequel le métal est un alliage d'aluminium ayant une teneur en silicium supérieure à 10 % en poids.

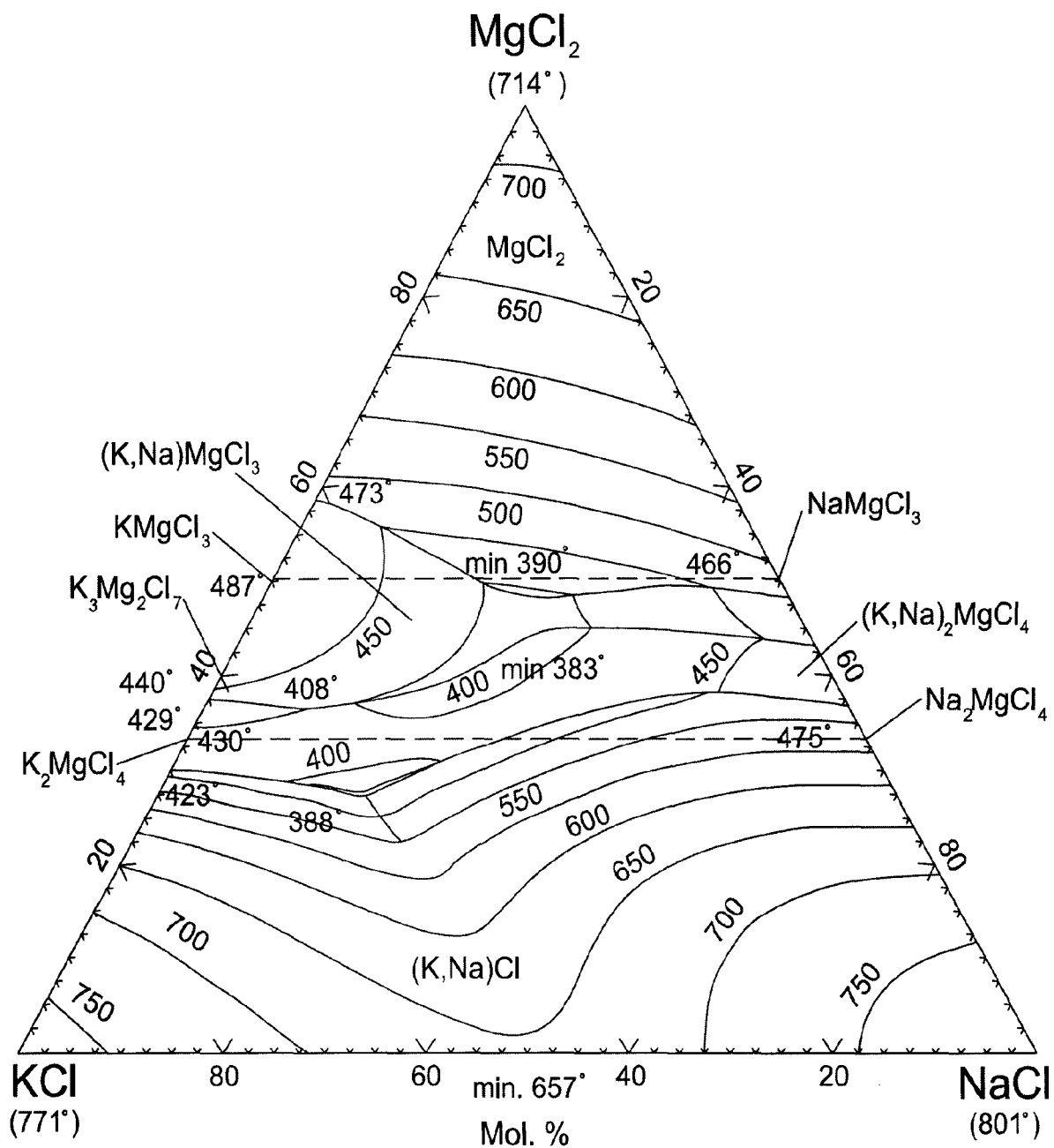


FIGURE 1
(PRIOR ART)

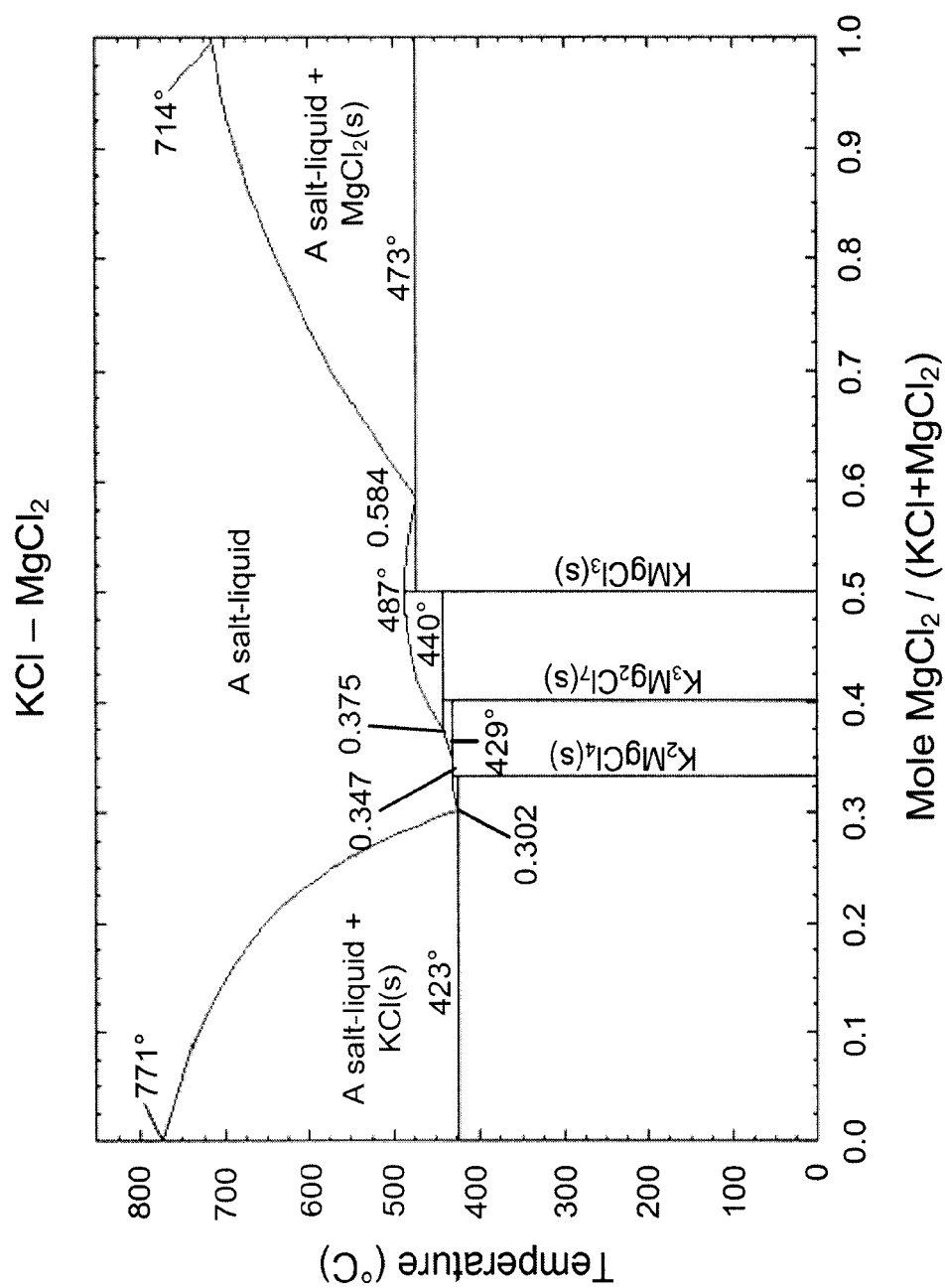


FIGURE 2
(PRIOR ART)

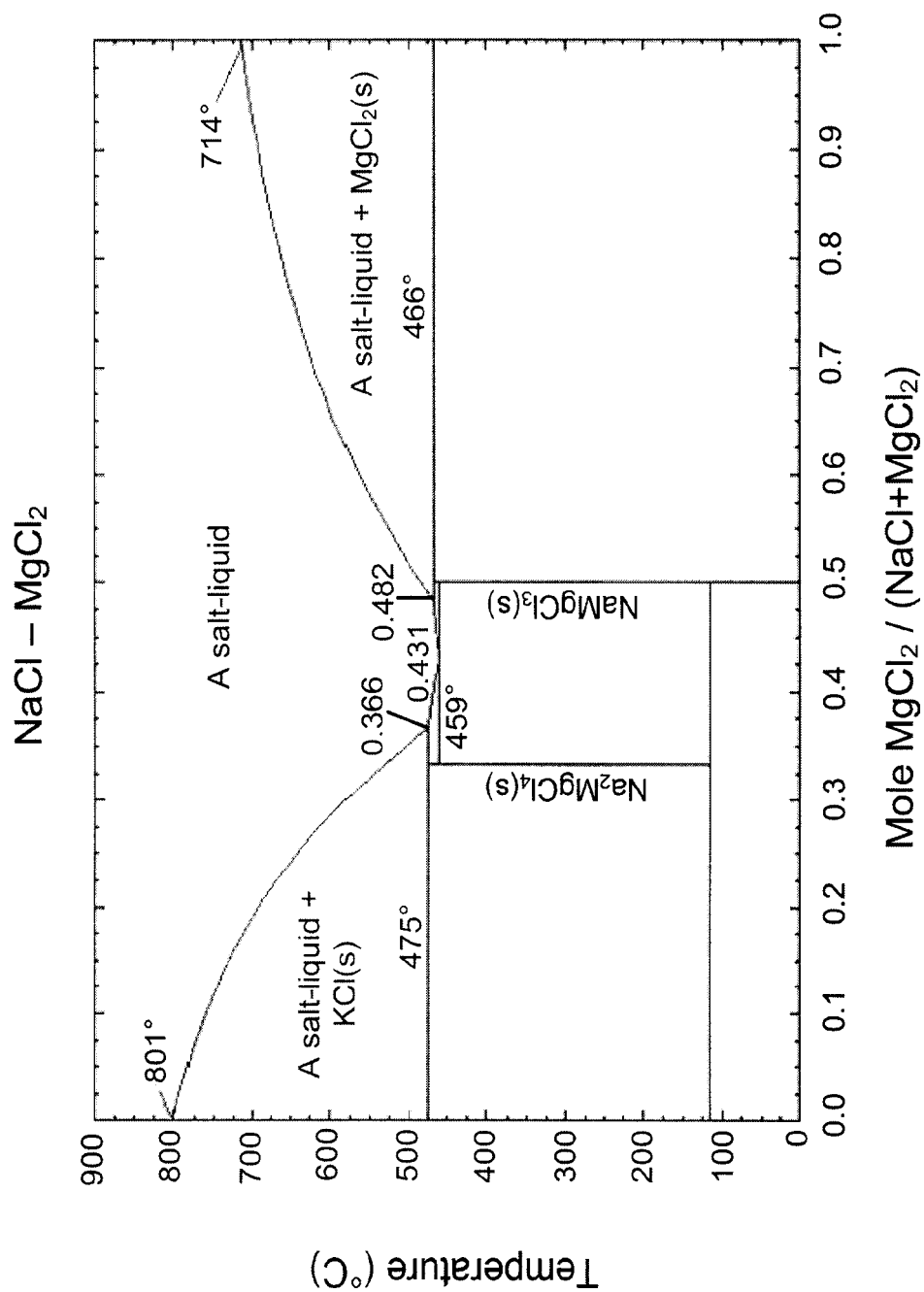


FIGURE 3
(PRIOR ART)

Efficiency of the withdrawal of calcium in a 2 kg 1100 aluminum alloy at 700 °C

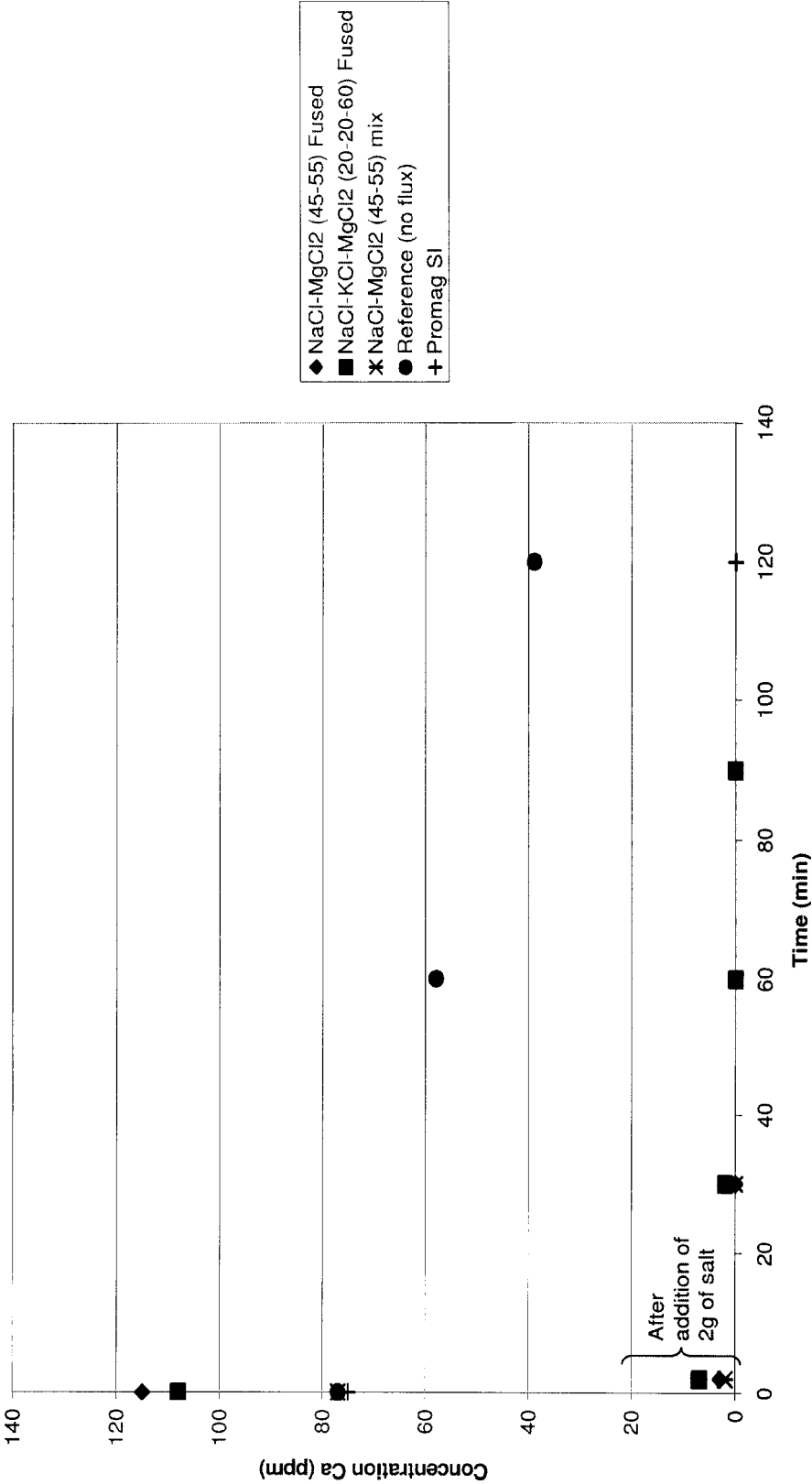


FIGURE 4

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- US 1377374 A [0009]
- US 1754788 A [0009]
- US 1519128 A [0009]
- US 2262105 A [0009]
- US 5405427 A [0009]
- US 4099965 A [0013]
- US 5427602 A [0014]

Non-patent literature cited in the description

- **DAVID H. DEYOUNG.** *Salt Fluxes for Alkali and Alkali and Alkaline Earth Element Removal from Molten Aluminum* [0010]
- **GRUZLESKI et al.** *The Treatment of Liquid Aluminum-Silicon Alloys*, 204-205 [0010]
- **ANTHONY COX.** Separation of Mg and Mn from Beverage Can Scrap using a Recessed-Channel Cell. *Journal of the Electrochemical Society*, 2003, vol. 150 (12), D200-D208 [0015]