

[54] **GRAIN REFINING COMPOSITIONS AND METHOD OF REFINING ALUMINUM THEREWITH**
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[63] Continuation-in-part of Ser. No. 359,756, May 14, 1973, abandoned.

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[51] **Int. Cl.** **C22b 21/06**
[58] **Field of Search** **75/93 R, 93 A, 93 E, 68 R, 75/135, 138, 93; 164/55, 56, 57**

References Cited			
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[57] **ABSTRACT**
Grain refining compositions for aluminium and aluminium alloys containing 15 – 85 percent by weight finely particulate Ti or high-Ti alloy, 2.5 – 70 percent by weight of an alkali metal complex fluoride and optionally a dense metal. The compositions are of density at least 2.4 g/cm³.

5 Claims, No Drawings

GRAIN REFINING COMPOSITIONS AND METHOD OF REFINING ALUMINUM THEREWITH

CROSS-REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of my application Ser. No. 359,756 filed 14th May, 1973 now abandoned.

This invention relates to improved grain refining compositions containing titanium metal.

It has previously been proposed to use metallic titanium as a grain refiner for aluminium and aluminium alloys. However, titanium metal and known grain refining compositions containing titanium metal suffer from a number of disadvantages.

Titanium metal dissolves too slowly in molten aluminium at normal commercial operating temperatures, with the result that in the time available between the addition of a grain refiner and casting solution of the titanium is incomplete. It has also been found that dissolution and hence recovery of titanium in the cast aluminium is influenced by the condition of the surface of the molten aluminium, the presence of dross or fluxing agents causing an exothermic reaction to take place and titanium recovery to be reduced. Some improvement can be effected if the titanium metal containing grain refiner is plunged and held below the surface of the molten aluminium. However, the plunging operation involves an additional step on the part of the foundryman, and there is a danger that the aluminium can become contaminated from the plunging tool.

It has now been found that titanium metal can be used efficiently as a grain refiner for aluminium if the titanium is used in the form of particles less than 500 microns in size, and is compacted with a complex salt to give a composition having a density such that the composition when added to molten aluminium will sink below the surface of the molten aluminium.

According to the present invention there is provided a composition for the grain refinement of aluminium or aluminium alloys comprising:

- i. 15 - 85 percent by weight metallic titanium and/or titanium alloy containing not less than 85 percent titanium,
- ii. 2.5 - 70 percent by weight of one or more of sodium borofluoride, potassium borofluoride, sodium titanofluoride, potassium titanofluoride, sodium zirconium fluoride and potassium zirconium fluoride, and
- iii. 0 - 46 percent by weight of a metal having a density greater than 4.5 g/cm³.

The content of said metal not exceeding the content of said titanium or titanium alloy, the content of component ii) being at least 12 percent of the content of said titanium or titanium alloy; said titanium and/or titanium alloy having a particle size not greater than 500 microns, and said composition having a density of at least 2.4 g/cm³.

The metal having a density greater than 4.5 g/cm³ may be, for example, manganese, copper, iron, nickel-chromium alloy or ferro-manganese. The actual metal and the amount used in the grain refining composition will be chosen such that the required specification of the cast aluminium or aluminium alloy is met. It is preferable to include at least 5 percent by weight of dense metal in the composition to ensure self-sinking even if, as may happen in manufacture, the tablet is imperfectly pressed.

Fluxing agents such as alkali metal or alkaline earth metal halides may be included in the grain refining composition. Carbonates may also be used. Examples of suitable fluxing agents are sodium chloride, potassium chloride, sodium fluoride, potassium fluoride, sodium aluminium fluoride and potassium aluminium fluoride. When used the content of the fluxing agent will not normally be more than 53 percent by weight.

The grain refining composition may be produced by any known method involving blending of the components and subsequent compaction to give a coherent mass having the required density. For example, the ingredients are mixed in a 1 ton capacity ribbon blade mixer and pressed to tablet form in a 70 ton BIPEL press.

If desired other material may be incorporated in the grain refining composition to aid in the production of a coherent mass, or the mass may be preheated.

When used to treat molten aluminium or aluminium alloys the amount added should be at a weight ratio of 0.005 percent to 0.2 percent (of Ti) by weight.

The following examples will serve to illustrate the invention.

In all the examples, the following test procedure was used:

The grain refining composition to be tested was made by mixing the ingredients together in a planetary action mixer, and portions of mixture were then tableted in a hydraulic tableting press at a pressure of 1,000 kg/cm². The resulting tablets had a weight of about 150 gms. and their density was then determined in the usual way.

The grain refining composition was tested for efficiency by adding the composition to molten LMO aluminium (99.8% AL), at a rate of 0.02 percent by weight of titanium. The LMO aluminium was held at 730°C.

Samples were taken and cast into preheated sillmanite test moulds two minutes before, ten minutes after and forty minutes after the addition of the composition.

The castings, 2.5 cm. diameter and 7.5 cm. high, were sectioned transversely 2.5 cm. from the base. The cut face of the base was then polished to 600 micron silicon carbide paper using water as lubricant and then etched using a mixed acid solution.

In all cases, the grain size evident after etching was visually much smaller in the samples poured after the addition of the grain refining composition but there was no substantial difference between the sample at ten minutes and the sample at forty minutes. Accordingly, grain size determinations were than made only on the test specimen before addition and that at forty minutes after addition.

The following are the grain refining compositions tested. Thereafter, the table gives the results of the test procedures just described. Percentages are by weight.

Example 1:		
	titanium (less than 500 microns)	70%
	potassium borofluoride	30%
Example 2:		
	titanium	30%
	manganese	20%
	potassium borofluoride	50%
Example 3:		
	titanium	40%

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Example 4:	manganese	40%
	sodium borofluoride	20%
	titanium	55%
	manganese	10%
	potassium borofluoride	11%
	potassium chloride	24%

RESULTS

RESULTS

Example No.	Density (gm/cc)	Grain Size before addition (microns)	Grain Size after addition (microns)
1	2.80	2600	500
2	2.89	3600	1500
3	3.49	2900	1000
4	2.73	2900	110

The following are further examples of grain refining compositions according to the invention (weight percent):

Example 5	Potassium chloride	5.0%
	Potassium borofluoride	38.0%
	Manganese	20.0%
	Titanium	37.0%

Example 6	Potassium chloride	5.0%
	Potassium borofluoride	28.0%
	Manganese	20.0%
	Titanium	47.0%

Example 7	Potassium chloride	19.0%
	Potassium borofluoride	14.0%
	Manganese	20.0%
	Titanium	47.0%

Example 8:	Potassium chloride	15.0%
	Potassium titanofluoride	10.0%
	Manganese	20.0%
	Titanium	55.0%

I claim:

1. A composition for the grain refinement of aluminum or aluminium alloys consisting essentially of

- i. 15 - 85 percent by weight of a material selected from the class consisting of metallic titanium and titanium alloys containing not less than 85 percent titanium,

- ii. 2.5 - 70 percent by weight of at least one component selected from the class consisting of sodium borofluoride, potassium borofluoride, sodium titanofluoride, potassium titanofluoride, sodium zirconium fluoride and potassium zirconium fluoride, and

- iii. 0 - 46 percent by weight of a metal having a density greater than 4.5 g/cm³,

the content of said metal not exceeding the content of said component i), the content of component ii) being at least 12 percent of the content of said component i). said component i) having a particle size not greater than 500 microns, and said composition having a density of at least 2.4 g/cm³.

2. A composition according to claim 1 wherein the metal of density greater than 4.5 g/cm³ is selected from the group consisting of manganese, copper, iron ferromanganese and nickel-chromium alloys.

3. A composition according to claim 1 and including up to 53 percent by weight of a compound selected from the group consisting of alkali and alkaline earth metal halides and carbonates as fluxing agent.

4. A method of treating molten aluminium and alloys thereof to effect grain-refinement, which comprises adding to said aluminium and alloys a composition consisting essentially of

- i. 15-85 percent by weight of a material selected from the class consisting of metallic titanium and titanium alloys containing not less than 85 percent titanium,

- ii. 2.5 - 70 percent by weight of at least one component selected from the class consisting of sodium borofluoride, potassium borofluoride, sodium titanofluoride, potassium titanofluoride, sodium zirconium fluoride and potassium zirconium fluoride, and

- iii. 0 - 46 percent by weight of a metal having a density greater than 4.5 g/cm³,

the content of said metal not exceeding the content of said component i), the content of component ii) being at least 12 percent of the content of said component i), said component i) having a particle size not greater than 500 microns, and said composition having a density of at least 2.4 g/cm³.

5. The method of claim 4 wherein the composition is added to the molten metal at a ratio of 0.005 to 0.2 percent of Ti by weight to said molten metal.

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