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Erklärungen gemäß Regel 4.17:

— *hinsichtlich der Berechtigung des Anmelders, ein Patent zu beantragen und zu erhalten (Regel 4.17 Ziffer ii)*

— *Erfindererklärung (Regel 4.17 Ziffer iv)*

Veröffentlicht:

— *mit internationalem Recherchenbericht (Artikel 21 Absatz 3)*

— *vor Ablauf der für Änderungen der Ansprüche geltenden Frist; Veröffentlichung wird wiederholt, falls Änderungen eingehen (Regel 48 Absatz 2 Buchstabe h)*

(54) Title: METHOD FOR FORMING A QUICKLY HARDENING, INORGANIC FOAM

(54) Bezeichnung : VERFAHREN ZUR BILDUNG EINES SCHNELL ERHÄRTENDEN, ANORGANISCHEN SCHAUMS

(57) Abstract: The invention relates to a method for forming a quickly hardening, inorganic foam on the basis of the reaction of two components: a) a solid component in the form of a reactive powder, which simultaneously has both framework-forming properties and pore-forming properties, and b) a liquid component in the form of an alkali metal silicate (water glass), wherein the reactive powder contains 45-65 wt% of aluminum oxide, 10-20 wt% of aluminum nitride, and 5-15 wt% of metal aluminum as essential constituents, the alkali metal silicate has a molar ratio of silicon oxide to metal oxide of 1.0-2.2, and the two components are mixed at a weight ratio of powder to liquid component of 0.5-2 to form a paste, from which a foam body having a bulk density less than 0.7 g/cm3 is then formed in an exothermic reaction in less than 10 minutes.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren zur Bildung eines schnell erhärtenden, anorganischen Schaums, basierend auf der Reaktion von 2 Komponenten: a) einer festen Komponente in Form eines reaktiven Pulvers, das gleichzeitig sowohl gerüstbildende als auch porenbildende Eigenschaften besitzt, und b) einer flüssigen Komponente in Form eines Alkalimetallsilikats (Wasserglas), wobei das reaktive Pulver als wesentliche Bestandteile 45 - 65 Gew.-% Aluminiumoxid, 10 - 20 Gew.-% Aluminiumnitrid und 5 - 15 Gew.-% metallisches Aluminium enthält, das Alkalimetallsilikat ein Molverhältnis Siliziumoxid zu Metalloxid von 1,0 - 2,2 hat, und die beiden Komponenten im Gewichtsverhältnis von Pulver zu flüssiger Komponente von 0,5 - 2 zu einer Paste gemischt werden, aus der sich dann in einer exothermen Reaktion in weniger als 10 Minuten ein Schaumkörper mit einer Rohdichte von weniger als 0,7 g/cm3 bildet.

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Method for forming a quickly hardening, inorganic foam

The invention relates to a process for forming a rapid-hardening inorganic foam based on the reaction of 2 components:

1) a solid component in the form of a reactive powder with at the same time structure-building and pore-forming properties, and
2) a liquid component in the form of an alkaline activation solution.

The invention further relates to a reactive powder for the preparation of a rapid-hardening inorganic foam by reaction with an alkaline activation solution.

Inorganic foam (mineral foam) is used as a thermally insulating, fire-resistant lightweight construction material. Rapid hardening of the foam is of advantage in the filling up of hollows in wall constructions, in the production of insulating boards and in the repair of the thermal insulation of industrial furnaces. In on-site processing it is advantageous to use a formula that requires mixing of only 2 components whenever possible. For thermal insulations of industrial furnaces the inorganic foam must be built up of components which offer high temperature resistance.

It is known to produce inorganic foam in the form of a geopolymeric foam by mixing at least 3 components,

1) reactive solids, containing SiO_2 and Al_2O_3 in sufficient amount and in reactive form (ashes, active clays, meta kaolin, pozzolans, slags etc.),
2) an alkaline activation solution that contains (apart from water) alkali hydroxides, alkali silicates, alkali alumi-

nates, alkali carbonates and alkali sulphates, either singly or in combinations thereof,

3) a pore-forming component (addition of foam, gas-forming reaction with aluminium powder or hydrogen peroxide).

[A. Buchwald: What are geopolymers? Current state of research and technology, the opportunities they offer, and their significance for the precast industry. BFT International 72, No. 7, 2006, 42-49]

US patent 3,396,112 describes a process of preparing inorganic foams by reacting water with a particulate mixture of aluminium and a dry, water-soluble alkali metal silicate, the particles of said mixture having a particle size of less than 10 mesh (2 mm), and the water content constituting from 30 to 75 weight percent of the total mixture. The weight ratio of said alkali metal silicate to said aluminium is from 15:1 to 1:9, and said alkali metal silicate has an alkali metal oxide to silica ratio of 2:1 to 1:4. According to the description of this patent (column 2, lines 55-58) commercially available aluminium powder or other forms of finely-divided aluminium having the corresponding particle size can be employed, and it is not necessary for the aluminium to be pure. Inert fillers such as silica, alumina, titania, and similar metal oxides can also be added (column 3, lines 8-11).

According to example 1 of this patent a foam product was generated by stirring 12.5 g water into a prefabricated dry mixture of 10 g aluminium powder (particle size less than 400 mesh) and 7.5 g solid sodium silicate (molar ratio $Na_2O:SiO_2 = 1 : 3.22$ and containing 17% by weight of water of hydration), while the formation of hydrogen and the connected generation of foam began after 15 seconds, and the mixture solidified after some minutes, connected with a temperature increase to 105 °C.

According to example 2 of this patent a dry mixture of 30 g of an aluminium dross (with a 70% metal content and a particle size of about 100 mesh) and 70 g of a sodium silicate (particle size about 200 mesh, molar ratio $Na_2O : SiO_2$ = 1 : 2, without water of hydration) is described to which was added 60 g of water; a foam formed within 30 minutes, which was then dried and hardened for 3 hours at 110 °C.

US patent 3,784,385 concerns the production of a refractory gas concrete having a thermal resistance up to 1,200 °C, consisting of 5 components:
1) a binder containing sodium silicate,
2) a finely ground filler in the form of a chrome-aluminium slag, in a weight percentage of 22 to 32 percent relative to the total weight when mixed with said binder,
3) addition of water,
4) a finely ground high-alumina refractory material and
5) a gas-forming agent in the form of sodium hydroxide and aluminium powder. In accordance with the description to this patent (column 2, line 67 to column 3, line 16) the mixture is heated to 38 - 42 °C for 3 - 5 hours, further treated for 10 hours in an autoclave at 170 - 180 °C, and finally stored for another 3 days at 20 °C.

US patent 4,133,691 describes a process for preparing an inorganic foam, having a molar ratio $SiO_2:Al_2O_3$ = 1:1 to 10:1, by mixing particulate metallic aluminium with an aqueous basic solution formed from a water-soluble alkali metal oxide, alkali metal hydroxide or alkali metal aluminate.

EP 1 180 504 B1 concerns a pore-forming, solidification-accelerating additive for binding agent-building materials, containing 50 - 90% Al_2O_3, 4 - 20% MgO, 0.5 - 15% SiO_2, 0.1 - 5% aluminium nitride, 0.1 - 10% metallic aluminium, 0.1 - 15% loss on ignition, and the main mineral constituents in the form of corundum (α-Al_2O_3) and spinel ($MgOx$-Al_2O_3), wherein the metallic aluminium particles are enveloped by mineral transition modifications from aluminium hydroxide ($Al_2O_3x3H_2O$) to α-Al_2O_3, and wherein the powder has a particle size of at least 90% smaller than 500 µm and a specific surface of at least 10 m^2/g. This additive is produced from a residue from the treatment of aluminium salt slag, by thermal dehydration and activation within a temperature range of 400 - 1000 °C. According to example 5, test 6, of this patent 450 g of "SEROX TK750" (powder, thermally activated at 750 °C, with a specific surface area of 45 m^2/g and a content of metallic aluminium of 3%) was mixed for about 1 minute with 400 g of liquid water glass. After a few minutes, the formation of gas in the paste began, resulting in the formation of foam and an increase in volume, connected with an increase of temperature to 76 °C. After a reaction time of 10 minutes the body was hardened so as to be mechanically stable, having a bulk density of 0.63 g/cm^3.

EP 0 576 254 A2 concerns a reactive non-metallic product with 7 - 25% aluminium nitride and 2 - 8% aluminium which is produced by treating aluminium dross and aluminium salt slag by washing out the chlorides (page 2, lines 53 - 55). A possible use of this product for the production of refractories or abrasives is described, but not for the formation of inorganic foam.

From the processing of aluminium dross with metal contents of 40 - 75% employing a plasma method (see US 4 960 460 A) the reactive powder "NOVAL" is known, which contains 53 - 65% aluminium oxide, 9 - 20% aluminium nitride and 5 - 12% metallic aluminium. The usability of "NOVAL" for the production of calcium aluminate, sodium aluminate, magnesium alu-

minate spinel, fused corundum, mineral fibers, as a cement supplement and as a sand-blasting medium is described, but not a possible suitability for the formation of inorganic foam.
[R. Breault, S.P. Tremblay, J. Lachance, Y. Huard: Market Opportunities for the ALCAN Plasma Dross Residues. Light Metals (1995) 823-827]
[R. Breault, D. Guay, G.Dubé, D.Legault, R.Morin, K. Annett, J. Bonneau: Aluminium Plasma Dross Treatment Process and Calcium Aluminate Production: Closing the loop with no residue. Light Metals (2000) 1183-1194]

The present invention seeks to provide a process of forming a rapid-hardening inorganic foam which, based on the reaction by 2 components, hardens in less than 10 minutes to give a stable body of foam, without requiring an external heat supply to achieve hardening, and, in addition, without the need of drying the foam body. In addition the mineral foam should have constituents which offer a high fire resistance. The present invention also seeks to provide a reactive powder for the formation of a rapid-hardening inorganic foam.

In one aspect there is provided a process of forming a process of forming a rapid-hardening inorganic foam based on the reaction of 2 components: a) a solid component in the form of a reactive powder with at the same time structure-building and pore-forming properties, and b) a liquid component in the form of an alkali metal silicate (waterglass), wherein the reactive powder comprises the essential constituents: 45 - 65% by weight aluminium oxide, 10 - 20% by weight aluminium nitride, 5 - 15% by weight metallic aluminium, and has a particle size of at least 90% by weight smaller than 1 mm, and the two components are mixed in a weight ratio of powder to liquid component of 0.5 to 2 to give a paste from which a foamed body with a bulk density of less than 0.7 g/cm^3 is then formed in an exothermal reaction within less than 10 minutes.

In another aspect there is provided a reactive powder for the forming of a rapid-hardening mineral foam by reaction with an alkaline activation solution, wherein said reactive powder comprises the essential constituents: 45 - 65% by weight aluminium oxide, 10 - 20% by weight aluminium nitride, and 5 - 15% by weight metallic aluminium, and has a particle size of at least 90% by weight smaller than 1 mm, preferably a particle size of at least 90% by weight smaller than 300 µm.

In another aspect there is provided a use of a powder which has been recovered from aluminium dross and which contains the essential constituents 45 - 65% by weight aluminium oxide, 10 - 20% by weight aluminium nitride, and 5 - 15% by weight metallic aluminium, as a reactive powder for the forming of an inorganic foam, particularly a lightweight construction material, and wherein said powder preferably has a particle size of at least 90% by weight smaller than 1 mm, more preferably smaller than 300 µm.

Since, in general, no heat supply from the outside is required for solidification of the inorganic foam, the process according to the present invention can, preferably, be carried out at room temperature, said room temperature generally being in the range from 15 °C to 30 °C, more particularly from 18 °C to 25 °C.

In the prior art, it has always been only the effect of metallic aluminium as a gas-forming component which has been considered in connection with the formation of inorganic foam. Surprisingly it has been found that it is possible to form a stable body of mineral foam with a bulk density of less than 0.7 g/cm^3, within less than 10 minutes and without the need of further drying, by reaction of a reactive powder having the main constituents 45 - 65% by weight aluminium oxide, 10 - 20% by weight aluminium nitride and 5 - 15% by weight metallic aluminium and preferably having a higher content of aluminium nitride than of metallic aluminium, with liquid alkali metal silicate. Furthermore the high content of aluminium oxide is of advantage for a high fire resistance of the inorganic foam. By means of x-ray diffraction analysis it can be seen that the mineral composition of the formed bodies of foam still contains some residual unreacted metallic aluminium while aluminium nitride cannot be detected any more, and that, hence, the aluminium nitride has reacted virtually completely.

These results were unexpected since, in accordance with US 3 396 112, example 1, when using pure aluminium powder in reaction with a solid water glass and addition of water the formation and solidification was completed already after a few minutes; however, according to US 3 396 112, example 2, the formation of the foam already took 30 minutes when using aluminium dross with a metal content of still 70% by weight.

The rapid forming of gas and solidification of the foam that was found to occur when using the powder according to the invention, being at the same time structure-building and pore-forming, in reaction with a liquid alkali metal silicate, is presumably based on the ammonia gas (from the reaction of $AlN + 2H_2O \Rightarrow AlOOH + NH_3$) that is released in addition to the hydrogen gas being formed (from the reaction $2Al + 4H_2O \Rightarrow 2AlOOH + 3H_2$), and the connected, additional formation of active aluminium hydroxide gel, which accelerates the formation of the framework structure by polymerization and consolidation of the alkali metal silicate in the way of a geopolymer.

A powder according to the invention, having structure-building as well as gas-forming properties, can be obtained, for example, in the recovery of aluminium from aluminium dross by mechanical crushing and classifying in the form of the grain size fractions of less than 1 mm, having the lowest content of aluminium, for example in the form of the screenings "CAI-ALON S-D" and the filter dust "CAI-ALON B-H", (manufacturer: CAST ALUMINIUM INDUSTRIES, Dubai, United Arab Emirates; Table 1). As can be seen from Table 1, the aforementioned powders of the present invention exhibit a BET surface area of 2.2 and 3.4 m^2/g, respectively.

As aforementioned, as the reactive powder according to the present invention, a powder may be used that has been recovered from aluminium dross, preferably a powder recovered from aluminium dross by mechanical size reduction and classification (particularly by screening, filtration or air classification)

According to the invention other reactive powders are also suitable, if they show the properties according to the invention, for example "NOVAL" obtained according to

US 4 960 460 or the non-metallic product according to EP 0 576 254 A2.

The BET surface area (specific surface area) of the reactive powder according to the present invention is preferably smaller than 10 m^2/g, more preferably smaller than 7.5 m^2/g, more particularly smaller than 5 m^2/g. BET surface area is determined according to ISO 9277 (1995), which replaces DIN 66131 (ISO 9277:1995; "Determination of the specific surface area of solids by gas adsorption using the BET method").

The reactive powder and the alkaline activation solution can of course also be used in combination with further mineral raw materials such as metakaolin, fly ash, slag, pozzolan, cement, lime, gypsum, sand, glass fibers, mineral fibers, if necessary adding further alkaline activation solution or water in an amount necessary for adjusting the consistency of the mixture to give a pasty consistency.

The present invention further relates to an inorganic foam or lightweight construction material that can be manufactured, or has been manufactured, using the process according to the present invention. More particularly, this foam or material is a thermally insulating or/and fire-resistant lightweight construction material.
The inorganic foam or lightweight construction material according to the present invention may also be present in the form of finished parts made therefrom, particularly insulating boards.

In the subsequent examples 1 - 6 (Table 3) the reactive powders "CAI-ALON S-D" and "CAI-ALON B-E" were mixed with an alkaline activation solution, based on the potassium water glass "SILIRIT M 110" (Table 2), in quantitative ratios of powder to water glass from 0.6 to 2 in a plastic mug and stirred to a paste at room temperature for approx. 30 sec, by using a spoon. The reaction temperature was measured by a thermometer introduced into the paste. After the formation and solidification of the foamed body, the bulk density of the body was determined by measuring its weight and outlines.

The results show that the consistency of the paste was readily spreadable with the examples 1 - 3, and the formation, the solidification and the bulk density of the foam were best; that is, at mixture ratios of solid to activation solution of a weight ratio of 1 : 1. The consistency of the paste in examples 4 and 6 was relatively thin, and in example 5 relatively thick, so that with regard to achieving a formation of an inorganic foam that is as rapid as possible, and with regard to obtaining an inorganic foam with a bulk density that is as low as possible, the best suitable mixing ratios between solid and activation liquid is considered to be in the range of 0.5 to 2. By x-ray diffraction analysis it could be confirmed that the mineral composition of the formed foam bodies still contained some residual metallic aluminium but no aluminium nitride, and that, hence, the aluminium nitride had reacted virtually completely, in contrast to the metallic aluminium.

Table 1: Reactive powders

		CAI-ALON S-D Screenings	CAI-ALON B-H Filter dust
Al_2O_3	[%]	49.9	54.9
AlN	[%]	12.6	15.0
Al metal	[%]	10.9	13.7
SiO_2	[%]	9.6	5.3
MgO	[%]	4.9	5.3
CaO	[%]	3.3	1.4
Na_2O	[%]	0.8	0.4
K_2O	[%]	0.6	0.9
Fe_2O_3	[%]	0.4	0.4
TiO_2	[%]	0.5	0.3
F	[%]	0.7	2.0
Cl	[%]	1.3	0.5
N	[%]	4.3	5.1
C	[%]	0.7	0.2
Moisture 105°C	[%]	0.1	0.1
Change in weight 1100°C	[%]	+7.7	+14.5
Specific surface area*	[m^2/g]	2.2	3.4
Particles <300μm	[%]	90	99
Main mineral constituents			
Corundum		+++	+++
Spinel		++	++
Aluminium Nitride		++	++
Aluminium Metal		++	++
Quartz		+	+

* BET surface area; determination using the BET method (ISO 9277)

Table 2: Alkaline Waterglass

Type:	SILIRIT M 110	
Supplier:	Cognis GmbH, D-40551 Düsseldorf / Germany	
Density	[kg/cm^3] (20 °C)	1600 - 1700
Viscosity	[mPas] (20 °C)	40 - 80
K_2O	[%]	27.80 - 31.00
SiO_2	[%]	22.20 - 23.00
Weight Ratio	[% SiO_2 / % K_2O]	0.74 - 0.80
Molar Ratio	[mol SiO_2 / mol K_2O]	1.16 - 1.26

Table 3: Examples

Example		1	2	3	4	5	6
CAI-ALON S-D	[g]	20	-	10	15	25	10
CAI-ALON B-H	[g]	-	20	10	-	-	10
SILIRIT M110	[g]	20	20	20	25	15	10
Water	[g]	-	-	-	-	-	10
Mixing ratio solid : water glass		1	1	1	0.6	1.66	2
Consistency of the paste		a)	a)	a)	b)	c)	b)
Time at start of reaction	[min]	1	1	1	1	1	4
Temperature max.	[°C]	126	132	126	130	110	93
Solidification	[min]	5	5	5	6	3	7
Bulk Density	[g/cm^3]	0.37	0.38	0.37	0.43	0.67	0.57

a) readily spreadable
b) relatively thin
c) relatively thick

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

The reference in this specification to any prior publication (or information derived from it), or to any matter which is known, is not, and should not be taken as, an acknowledgement or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

Whilst the present invention has been hereinbefore described with reference to a particular embodiment, it will be understood that numerous variations and modifications will be envisaged by persons skilled in art. All such variations and modifications should be considered to fall within the scope of the invention as broadly hereinbefore described and as hereinafter claimed.

The claims defining the invention are as follows:

1. A process of forming a rapid-hardening inorganic foam based on the reaction of 2 components:
a) a solid component in the form of a reactive powder with at the same time structure-building and pore-forming properties, and
b) a liquid component in the form of an alkali metal silicate (waterglass), wherein
the reactive powder comprises the essential constituents:
45 - 65% by weight aluminium oxide,
10 - 20% by weight aluminium nitride,
5 - 15% by weight metallic aluminium, and
has a particle size of at least 90% by weight smaller than 1 mm, and the two components are mixed in a weight ratio of powder to liquid component of 0.5 to 2 to give a paste from which a foamed body with a bulk density of less than 0.7 g/cm^3 is then formed in an exothermal reaction within less than 10 minutes.

2. The process in accordance with claim 1, wherein the components are mixed for maximum 5 minutes, preferably maximum 1 minute.

3. The process in accordance with claim 1 or 2, wherein the components are mixed without the supply of heat from the outside, preferably at room temperature, to give a paste.

4. The process in accordance with any one of claims 1 to 3, wherein the alkaline activation solution is an alkali metal silicate solution having a molar ratio of silicon oxide to metal oxide of 1.0 to 2.2.

5. The process in accordance with any one of the preceding claims, wherein the reactive powder has a weight ratio of aluminium nitride to metallic aluminium of greater than 1.

6. The process in accordance with any one of the preceding claims, wherein the reactive powder has a particle size of at least 90% by weight smaller than 300 µm.

7. The process in accordance with any one of the preceding claims, wherein the reactive powder is mixed with a liquid potassium water glass, having a molar ratio (SiO_2 : K_2O) of 1.16 to 1.26.

8. The process in accordance with any one of the preceding claims, wherein the reactive powder and the alkaline activation solution are used in combination with other mineral raw materials, such as metakaolin, fly ash, slag, pozzolan, cement, lime, gypsum, sand, glass fibers, mineral fibers, and that, if necessary, further alkaline activation solution or water is added in a quantity necessary for adjusting a pasty consistency of the mixture.

9. The process in accordance with any one of the preceding claims, wherein the BET surface area of the reactive powder is smaller than 10 m^2/g, preferably smaller than 7.5 m^2/g, more preferably smaller than 5 m^2/g.

10. The process in accordance with any one of the preceding claims, wherein as the reactive powder a powder is used which has been recovered from aluminium dross, preferably a powder recovered from aluminium dross by mechanical size reduction and classification.

11. A reactive powder for the forming of a rapid-hardening mineral foam by reaction with an alkaline activation solution, wherein said reactive powder comprises the essential constituents:
45 - 65% by weight aluminium oxide,
10 - 20% by weight aluminium nitride, and
5 - 15% by weight metallic aluminium, and
has a particle size of at least 90% by weight smaller than 1 mm, preferably a particle size of at least 90% by weight smaller than 300 µm.

12. The reactive powder in accordance with claim 11, wherein the weight ratio of aluminium nitride to metallic aluminium is greater than 1.

13. The reactive powder in accordance with any one of claims 11 or 12, wherein the BET surface area of the reactive powder is smaller than 10 m^2/g, preferably smaller than 7.5 m^2/g, more preferably smaller than 5 m^2/g.

14. The reactive powder in accordance with any one of claims 11 to 13, wherein it has been recovered from aluminium dross, preferably by mechanical size reduction and classification.

15. Use of a powder which has been recovered from aluminium dross and which contains the essential constituents
45 - 65% by weight aluminium oxide,
10 - 20% by weight aluminium nitride, and
5 - 15% by weight metallic aluminium,
as a reactive powder for the forming of an inorganic foam, particularly a lightweight construction material, and where-

in said powder preferably has a particle size of at least 90% by weight smaller than 1 mm, more preferably smaller than 300 µm.

16. The use according to claim 15, wherein the reactive powder is reacted with a liquid component in the form of an alkali metal silicate, to form the inorganic foam.

17. The use according to claim 15 or 16, wherein the weight ratio of aluminium nitride to metallic aluminium in the powder is greater than 1.

18. The use according to any one of claims 15 to 16, wherein the BET surface area of the reactive powder is smaller than 10 m^2/g, preferably smaller than 7.5 m^2/g, more preferably smaller than 5 m^2/g.

19. Inorganic foam or lightweight construction material which can be obtained by a process according to any one of claims 1 to 10, preferably a thermally insulating or/and fire-resistant lightweight construction material.

20. The inorganic foam or lightweight construction material according to claim 19, wherein it is present in the form of finished parts, particularly in the form of insulating boards.