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REFRACTORY FELT

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ABSTRACT OF THE DISCLOSURE

A refractory felt and method of making same comprising preparing a web of substantially insoluble refractory mineral fibers having a large specific surface and treating the web by continuously rolling at elevated temperatures such that the fibers sinter together where they contact.

BACKGROUND

For many years the refractory industry has produced refractory fibers. Most of these fibers were obtained by melting refractory materials, for example, dehydrated kaolinic clay, sometimes enriched with alumina, followed by blowing a jet of the molten refractory into fibers by techniques similar to those used in the glass, slag, and rock-fiber industries.

Another technique, which is apparently less widespread, consists in concentrating a solution of an aluminum salt, generally the oxychloride, until a viscous mass is obtained which is capable of being converted into fibers by any suitable means, such as blowing, centrifuging, and so on.

Fibers made according to these two processes are marketed either in bulk or in the form of webs or felts. Webs are obtained by sucking fibers as they are produced onto grids. This technique has a serious drawback: The various layers of fibers forming the web are not joined or secured together. Hence, these webs tend to cleave, preventing their being used in numerous applications.

In order to overcome this drawback, webs and even felts can be manufactured with fibers orientated in all directions, the various techniques consisting of dispersing the unitary fibers in water and gathering these dispersions on a filter to form webs or felt. These methods have two disadvantages; they partly destroy the fibers and they require the addition of organic binder materials which burn away at elevated temperatures. The good cold bending and tensile strengths of the webs and felts largely disappear when the temperature exceeds 350° C. and they become brittle and friable.

THE INVENTION

The process of this invention consists in manufacturing refractory felt from fiber such as those fibers, for example, disclosed in our U.S. application Ser. No. 720,324, filed Apr. 10, 1968, entitled "Catalytic Mineral Fibers and Their Preparation." That application discloses substantially insoluble refractory mineral fibers having a large specific surface and containing more than 70 percent, preferably 80 to 95 percent, of alumina, the remainder being constituted by at least one other refractory, for example, a catalytic oxide and a method of making them. The web made from these fibers is treated for a period of from 0.5 to 2 hours by compressing by rolling at a pressure less than 20 grams per square centimeter and at a temperature between 1,200 and 1,300° C.

This treatment results in a linear shrinkage of the fibers from 10 to 15 percent and to slight sintering of the fibers to one another. The cohesion of the felt is thus

considerably increased and its mechanical properties are retained to the temperature known as the limit temperature of use, which conventionally is that temperature to which the felt can be subjected for 24 hours without shrinking more than 3 percent. The limit temperature of use for felts prepared according to our invention reaches and exceeds 1,500° C.

It should be observed that the sintering takes place only because the fibers made, for example, by the process of our aforementioned U.S. application Ser. No. 720,324 have a large specific surface.

The process according to this invention is suitable for continuous manufacture, as can be seen from the following more detailed description.

An aqueous solution containing at least aluminum oxychloride, silica, and optionally soluble refractory oxide salts, batched to enable the desired final chemical composition is concentrated to a viscosity of 150 to 200 poises.

By any known means, for example, by centrifuging this mother solution at least than 100° C., blowing, and gathering by suction, a continuous web is produced the width of which may exceed 1 meter. Its thickness may preferably range from 50 to 150 mm. The web is composed of intermingled fibers the diameter of which is preferably smaller than 10μ and the length of which may range from a few centimeters to more than one meter. This primary web of soluble fibers should preferably have an apparent specific gravity between 0.02 and 0.04.

After drying, this primary web is continuously treated in a current of water vapor at 350° C. ±20° C. for from 2 to 12 hours, so as to obtain a secondary web of fibers having a large specific surface, preferably greater than 70 square meters per gram, and composed of insoluble oxides. This treatment is accompanied by homogeneous linear shrinkage of from 20 ot 25 percent.

This secondary web is then subjected to continuous rolling at low pressure for from 0.5 to 2 hours at a temperature in excess of 1200° C. and preferably from 1,200 to 1,300° C.

The further shrinkage and the incipient sintering effected by this treatment increase the apparent density of the web and impart to it remarkable tensile strength up to temperatures, such as 1,500° C. and higher.

The apparent density of the final web thus obtained increases with the rolling pressure. It amounts to about 0.07 to 0.15 for a pressure between 2 and 20 grams per square centimeter. It may be lower than 0.07 for lower rolling pressures.

Among the most desirable products manufactured by the process of this invention, are those which have the following final compositions by weight:

	Percent
(1) Limit temperature of use: 1,500° C.:	
Al_2O_3	80–95
SiO_2	5–20
(2) Limit temperature of use: at least 1,600° C.:	
Al_2O_3	80–90
SiO_2	5–15
Cr_2O_3	3–10

Nevertheless, within the scope of this invention the additions, such as silica and chromium oxide, may also be replaced at least partially by other oxides, such as zirconium and/or thorium oxides.

The process according to this invention for making refractory felts may be a continuous process, for example, a continuous secondary web may be continuously moved through an elongated furnace in which the web is heated to a temperature in excess of 1,200° C. and moved under refractory rollers which are secured within the furnace.

The following example relates to the manufacture of a refractory felt of type 2.

Example

A solution of aluminum oxychloride with a density of 1.33, and with a weight ratio of Al_2O_3/Cl equal to 2.45, is prepared. Its pH is brought to a value between 3 and 5 by adding acetic acid. To this solution are added (1) a silica sol containing 40 percent by weight of silicic oxide, in an amount such that the weight ratio of SiO_2/Al_2O_3 will be equal to 0.12, and (2) a solution of chromic anhydride, CrO_3, in an amount such that the final weight ratio of Cr_2O_3/Al_2O_3 will be equal to 0.05. This solution is evaporated at 50° C. at a pressure of 65 mm. Hg to a viscosity of 150 poises, measured at 18° C. It is centrifuged at 3,500 r.p.m. through orifices of a diameter of 0.2 mm. in an atmosphere having a relative humidity of less than 30 percent, at 35–40° C. A primary web having an apparent density of 0.025 is thus obtained.

After drying at 80° C. for 6 hours, the primary web is treated at 350° C. for 8 hours in a current of water vapor at atmospheric pressure. The hydrates, chlorides, and oxychlorides are entirely converted into insoluble oxides. The linear shrinkage in this stage amounts to 20 percent.

Finally, this secondary web is subjected in a roller furnace to heat treatment for 45 minutes at 1,250° C. under a mechanical pressure of 5 grams per square centimeter. A further linear shrinkage of 12 percent is effected, together with incipient sintering of the fibers to one another.

The final felted web has the following characteristics:

Chemical analysis, by weight:	Percent
Al_2O_3	81.6
SiO_2	14.4
Cr_2O_3	4

Apparent density: 0.12 gm./cc.
Tensile strength: 800 to 1,000 grams per square centimeter
Post-shrinkage:
1.5 percent after 24 hours at 1,500° C.
3 percent after 24 hours at 1,600° C.
(Limit temperature of use)

Thermal conductivity in Kcal./m./m.²/° C./h.:

At 200° C. (mean temperature)	0.06
At 400° C.	0.08
At 600° C.	0.14
At 800° C.	0.16

Having thus described our invention with the particularity required by the patent laws, what we desire to have protected by Letters Patent are as follows:

We claim:

1. A method of making a refractory felt comprising at least 70 percent alumina, by weight, comprising the steps of:
(1) preparing a web of substantially insoluble refractory mineral fibers having a large specific surface
(2) treating the web by compressing at temperatures sufficient to enable sintering such that fibers sinter together where they contact.

2. A method according to claim 1 in which the fibers are treated for a period of about 0.5 to 2.0 hours by continuously rolling at a pressure less than 20 grams per square centimeter at a temperature in excess of 1,200° C.

3. A method according to claim 2 in which the fibers are treated at a temperature between 1,200 and 1,300° C.

4. A method according to claim 2 in which the specific surface of the fibers is greater than about 70 square meters per gram.

5. A method according to claim 4 in which the refractory fibers comprise in addition to alumina, silica and at least one other refractory oxide.

6. A method according to claim 4 in which the refractory fibers contain in addition to alumina at least one catalyst.

7. A method according to claim 4 in which the refractory fibers comprise from 80 to 95 percent, by weight, alumina.

8. A method according to claim 4 in which the webs are treated by continuously moving them through a furnace in which they are heated and rolled.

9. A refractory felt made according to the method of claim 4 whereby the felt has an apparent density less than 0.15 gm./cc. and a limit temperature of use in excess of 1,500° C.

10. A method of making a refractory felt comprising at least 70 percent alumina, by weight, comprising the steps of
(1) forming an aqueous solution comprising aluminum oxychloride
(2) concentrating the solution
(3) forming fibers from the solution and gathering them in a primary web of soluble fibers
(4) drying the web and then treating it in water vapor at elevated temperatures to form a secondary web of insoluble fibers
(5) treating the secondary web for a period of 0.5 to 2.0 hours by continuously rolling at a pressure less than 20 grams per square centimeter at a temperature in excess of 1,200° C.
(6) recovering a felt in which fibers are sintered together where they contact.

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