United States Patent [19]
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[11] **3,891,595**
[45] **June 24, 1975**

[54] **FRICTION MATERIAL**

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[22] Filed: **Aug. 24, 1973**

[21] Appl. No.: **391,515**

[30] **Foreign Application Priority Data**
Aug. 30, 1972 United Kingdom 40189/72

[52] **U.S. Cl.** **260/38**; 106/38; 260/DIG. 39
[51] **Int. Cl.** **C08g 51/04**
[58] **Field of Search** 260/DIG. 39, 38; 106/36

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[57] **ABSTRACT**

Improved friction materials comprising a synthetic metal oxide fibre and a binder. The most useful fibres are polycrystalline alumina, alumina/silica or zirconia, and especially those made by fibrising an aqueous solution of a compound of the metal containing a water-soluble organic polymer and having a viscosity above 1 poise, drying the fibre and heating. Fibres of other oxides or mixed oxides may be used. The materials may include additives (lubricants, fillers, and metals) and are useful in brake units, in which they have advantages over conventional (cotton and asbestos) materials in their heat stability and reduced tendency to "fade" in service.

19 Claims, No Drawings

FRICTION MATERIAL

This invention relates to friction materials and particularly, though not exclusively, to friction materials usable for brake and clutch surfaces.

Conventional friction materials consist normally of cotton or asbestos fibres impregnated with a resin and made up into a variety of forms such as segments, cones, discs, rollers and so forth, which forms may be rigid or flexible. Cotton is used largely for light duties involving relatively low temperatures at the friction interface, whereas asbestos is used for heavier duties and consequent higher temperatures. As interface temperature increases, however, the friction coefficient falls, leading to brake "fade." In addition, a proportion of the asbestos may be liberated from the friction material as the latter wears and can produce a significant atmospheric pollution problem, for example in the case of automobiles used in towns.

According to the present invention there is provided a friction material comprising a synthetic metal oxide fibre and a binder. Synthetic inorganic fibres can be produced with controlled physical properties, which is an advantage in making friction materials with reproducible properties.

The fibre may be used in unwoven or woven form for example loose staple, papers, felts, mats or cloths.

We have found that synthetic inorganic fibres comprising one or more polycrystalline refractory metal oxides are especially suitable, as their physical properties make them capable of withstanding extremes of temperature conditions without serious loss of strength and they can be readily produced at the fine diameters and shot-free quality preferred. Vitreous fibres tend to devitrify under severe conditions of temperature or under the influence of other aggressive conditions. We prefer to use single metal oxides, double metal oxides or mixtures of oxides; these include thoria; urania; rare earth oxides; yttria; titania; chromia; magnesia; zinc oxide; calcia; double oxides of alkaline earth oxides and alumina, zirconia or chromia, for example $BaO/6Al_2O_3$, BaO/ZrO_2, CaO/Cr_2O_3, MgO/Al_2O_3, MgO/Cr_2O_3, SrO/ZrO_2; and other double oxides, for example Al_2O_3/TiO_2; CoO/Al_2O_3; NiO/Al_2O_3; ThO_2/ZrO_2; ZnO/Al_2O_3. We especially prefer to use polycrystalline alumina, alumina/silica or zirconia fibres prepared for example as disclosed in our co-pending United Kingdom Patent Applications Nos. 36,693/72; 12,088/72; 4,369/71 and 29,909/70, as fibres thus prepared have a high coefficient of friction, are hard and have other desirable properties as hereinafter described. These disclosures are incorporated herein by reference.

As disclosed in these applications alumina or zirconia fibres are formed by fibrising a composition having a viscosity of greater than 1 poise comprising an aqueous solution of a metal compound for example an oxychloride, basic acetate, basic formate or nitrate of aluminium and/or zirconium, and a minor proportion of a water-soluble organic polymer especially polyethylene oxide, polyvinyl alcohol (and, when alumina/silica fibres are requires, a water-soluble organic silicon compound such as polysiloxane), drying the fibre formed and heating to decompose the metal compound to oxide and to decompose the polymer. Heating in the presence of steam is often preferred.

Fibrising is preferably carried out by a blowing process which comprises extruding the fibrising composition through one or more apertures into at least one converging gas stream having a component of high velocity in the direction of travel of extruded composition. The dimensions and shape of the said aperture may vary widely. We prefer to use an aperture having at least one dimension larger than 5 microns and smaller than 500 microns. The gas stream is preferably air, more preferably air at ambient temperature. It is convenient to employ two streams of gas which converge at or near the point where the composition is extruded from the aperture; preferably the angle between the converging gas streams is from 30° to 60°. At least part of the water in the composition is removed by the gas stream, and the rate of removal may conveniently be controlled by mixing the gas with the water vapour, for example air at a relative humidity of greater than 80 percent may be used. The velocity of the air stream may be varied over wide limits, but we prefer to use velocities in the region of 200 to 1,500 feet per second. The pressure employed to extrude the composition through the apertures will depend on the viscosity of the composition and on the desired rate of extrusion. We find that pressures from 16 to 100 pounds per square inch absolute are convenient for compositions having viscosities up to about 100 poise.

The average diameter and diameter distribution of the fibres is important; fine fibre diameters and relatively narrow diameter distributions are preferred. An average fibre diameter from 0.5 to 10 microns is especially preferred; a diameter distribution which ensures that the fibres contain not greater than 30 percent by weight of fibres of greater diameter than 5 microns, or not greater than 20 percent by number of fibres of greater than 5 microns is also especially preferred. Alumina or zirconia fibres available under the Trade Mark of "Saffil" are especially useful as such fibres have these properties.

Growth of the crystallites in the fibres on storage or during use is preferably prevented, for example by the presence in the fibre of a stabiliser, for example in the case of zirconia fibres a minor proportion, for example 1 to 10 percent of one or more of alkaline oxides, yttria, rare earth oxides, alumina, thoria or hafnia, and in the case of alumina fibres a minor proportion of silica.

The amount of shot in the fibres, that is, material of a non-fibrous nature, should be as low as possible; shot content less than 5 percent by weight is preferred.

Alumina fibres are especially useful in preferred embodiments of the invention. Alumina exists in a number of phase forms and transformation of one to the other depends upon the temperature to which the alumina is subjected; while not wishing to be restricted to any particular theory it is believed that this property of alumina gives an advantage over many other inorganic fibres used in friction material. Thus in use, the friction materials according to the invention are subjected to a temperature which depends upon the degree of friction produced at a braking interface. At a temperature greater than about 700°C non-crystalline alumina is converted, at least partially, to a transitional alumina (for example "eta" alumina). At higher temperatures, for example greater than 950°C, "alpha" alumina is formed. The transitional and alpha aluminas consist of small crystallites, for example of the order of 100 Angstroms, which, although hard and friction-generating, are not gritty so that they do not accelerate the wear of the braking counter-surface. In addition, the hardness of alpha alumina is greater than that of transitional alumina which is in turn harder than non-crystalline alumina. It is believed therefore that as the friction temperature increases the coefficient of friction between the braking surface increases. Thus a braking action involving friction materials according to the invention is less subject to fading.

The temperatures at which the non-crystalline alumina is converted to a transitional alumina or a transitional alumina is converted to alpha alumina may be varied within useful limits by suitable modification of the composition of the alumina fibres and/or the methods used to make them. Methods used to modify the composition of alumina fibres are disclosed in the patent applications hereinbefore referred to. Thus a desirable degree of control over the effect of temperature on coefficient of friction may be achieved by a suitable modification of the alumina fibre composition or the use of suitable blends of alumina fibre.

The binder used in the friction material according to the invention is preferably an organic binder, for example a thermosetting resin or a natural or synthetic rubber. The most preferred binder is one based on a phenol-formaldehyde resin. Combinations of binders may also be used.

The friction materials according to the invention may comprise further materials to achieve any particular desired combination of properties. Thus lubricants, for example litharge, graphite or molybdenum disulphide; fillers, either inert or friction-augmenting, for example ground barytes, perlite, corundum, borax or clays; metals such as zinc, copper, brass, lead or iron, conveniently in the form of wires, turnings or powders for example as scavengers may be incorporated as desired.

The relative proportions of metal oxide fibre, binder and other ingredients of the friction material are chosen as normally in the art to give the properties required for the duty to be performed. Normally the binder is present in the range 15 to 35 percent by weight of the total, the metal oxide fibre in the range 40 to 85 percent by weight of the total and other ingredients from 0.1 to 25 percent by weight of the total.

The friction materials according to the invention are prepared by any convenient process known in the art. The fibre is incorporated into the binder and the mixture shaped into the desired end form for example by dry processing, sheeter processing or processes suitable for the incorporation of woven fibre into the binder. Wet board processing is especially preferred for loose fibre; in this process the weighed ingredients except the binder are dispersed for example by adding to a beater or hydrapulper with a quantity of water. The wet mixed pulp is then further diluted and felted for example by feeding to a paper-making machine to be formed into a blanket of suitable thickness. This felted product is then saturated in a liquid binder, for example a solution of a phenolic resin in an evaporable solvent, shaped and solidified usually by drying. For fibre in the form of mats, blankets and papers, the beating stage may be omitted.

The friction materials according to the invention may be fabricated or formed into any shape suitable for the application in which it is to be used. Thus they may be formed into segments or discs for use as brake linings or clutch facings.

The invention is illustrated by but not limited to, the following Examples.

EXAMPLE 1

30 grams of Saffil alumina fibre blanket (ex ICI Ltd.) was shredded into approximately 1 cm pieces, added to about 250 ml of water and dispersed by means of a high speed laboratory stirrer. The dispersed fibres were separated off, dried and mixed with 10 grams of a heat-setting liquid phenolic resin (R6741 ex BXL) dissolved in 100 ml of acetone. The acetone was removed by applying vacuum. The dry mix was then slightly moistened with water and compressed into a small block in a hydraulic press at 4,500 pounds line pressure. The compressed block was dried at 90°C and cured at 130°C for several hours (e.g., over night).

Friction/wear tests were carried out on the block using the rubber wear tests defined by BS.903 Part A9 (Method A) and ASTM D394.47 (Method A) using a Croydon abrasion tester. Abrasive paper (600C grade) was used on the disc and a force of 3.6 kg (approximately equal to 1.8 kg/cm^2) was used to hold the sample against the disc.

Comparative results were obtained using fine glass fibre, asbestos fibre and an alumino-silicate fibre in place of the alumina fibre in the test block. The results obtained are shown in Table 1.

Table 1

Fibre Sample	Coefficient of Friction (μ)	Wear (Asbestos=1)
"Saffil" alumina	0.59	0.08
Fine glass	0.50	0.08
Alumino-silicate	0.46	1.00
Asbestos	0.51	1.00

EXAMPLE 2

Example 1 was repeated using Saffil zirconia fibre in place of the alumina fibre. The results were: Coefficient of Friction (μ) 0.51; wear (asbestos=1) 0.04.

EXAMPLE 3

Test blocks were made as in Examples 1 and 2 using Saffil alumina fibre or Saffil zirconia fibre, but in addition to the fibre a filler a filler was added to the wet mix of fibre and resin solution. Ground kaolin and perlite were used as fillers and the results of friction wear tests carried out as described in Example 1 are shown in Table 2.

Table 2

Composition (parts by weight)		Coefficient of Friction (μ)	Wear Rate
"Saffil" zirconia	60	0.45	0.05
Perlite	20		
Resin	20		
"Saffil" alumina	60	0.48	0.20
Perlite	20		
Resin	20		
"Saffil" zirconia	55	0.53	0.40
Kaolin	33		
Resin	12		
"Saffil" alumina	55	0.49	0.30
Kaolin	33		
Resin	12		

What I claim is:

1. A friction material comprising from 40 to 85 percent by weight of a synthetic inorganic polycrystalline refractory metal oxide fibre having an average fibre diameter of from 0.5 to 10 microns having a shot content of less than 5 percent by weight,

and from 15 to 35 percent by weight of a binder.

2. A friction material as claimed in claim **1** wherein the fibre comprises thoria, urania, a rare earth oxide, yttria, titania, chromia, magnesia, zinc oxide, calcia or a double oxide of an alkaline earth oxide and alumina, zirconia or chromia, or mixtures thereof.

3. A friction material as claimed in claim **1** wherein the fibre is a polycrystalline alumina fibre.

4. A friction material as claimed in claim **1** wherein the fibre is a polycrystalline zirconia fibre.

5. A friction material as claimed in claim **3** wherein the fibre is formed by fibrising a composition having a viscosity greater than 1 poise comprising an oxychloride, basic acetate, basic formate or nitrate of aluminium and a minor proportion of a water-soluble organic polymer, drying the fibre and heating to form the oxide of aluminium and to decompose the polymer.

6. A friction material as claimed in claim **4** wherein the fibre is formed by fibrising a composition having a viscosity greater than 1 poise comprising an oxychloride, basic acetate, basic formate or nitrate of zirconium and a minor proportion of a water-soluble organic polymer, drying the fibre and heating to form the oxide of zirconium and to decompose the polymer.

7. A friction material as claimed in claim **5** wherein the water-soluble organic polymer is polyethylene oxide, polyvinyl-alcohol or polyvinylpyrrolidone.

8. A friction material as claimed in claim **3** wherein fibrising is effected by extruding the composition through one or more apertures into at least one converging gas stream having a component of high velocity in the direction of travel of the extruded composition.

9. A friction material as claimed in claim **8** wherein the gas is air at a relative humidity of greater than 80 percent.

10. A friction material as claimed in claim **1** wherein the metal oxide fibre has a fibre diameter distribution such that the fibres contain not greater than 30 percent by weight of fibres of greater diameter than 5 microns.

11. A friction material as claimed in claim **4** wherein zirconia fibre is stabilised with a minor proportion of an alkaline earth oxide, yttria, a rare earth oxide, alumina, thoria or hafnia or mixtures thereof.

12. A friction material as claimed in claim **3** wherein the alumina fibre is stabilised with a minor proportion of silica.

13. A friction material as claimed in claim **3** wherein the alumina is transitional alumina.

14. A friction material as claimed in claim **1** wherein the binder is based on a phenol-formaldehyde resin.

15. A friction material as claimed in claim **1** comprising a lubricant selected from litharge, graphite or molybdenum disulphide.

16. A friction material as claimed in claim **1** comprising a filler selected from ground barytes, perlite, corundum, borax or a clay.

17. A friction material as claimed in claim **1** comprising a metal in the form of wires, turnings or powders selected from zinc, copper, brass, lead or iron.

18. A process for preparing a friction material as claimed in claim **1** wherein a synthetic metal oxide fibre is incorporated into a binder and the mixture shaped into a desired end form.

19. A process as claimed in claim **18** wherein the friction material is prepared by wet board processing, comprising the steps of dispersing the fibre, felting the dispersed fibres, saturating the felted fibres in a liquid binder, shaping and solidifying.

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