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HEAT RESISTANT PRODUCT AND METHOD

The invention relates to a heat resistant product and a method for producing the product.

5 There are many applications where heat insulation and in particular fire resistance is required. Important examples include fire walls and doors for buildings and ships, ceilings, walls and internal panels of trains and other vehicles and heat shields for high performance
10 aircraft, for example leading edges of wings, noses and cones.

Various coating materials and the like have been proposed in the past to promote heat insulation in these applications but these have been found to be inefficient
15 and/or non-cost effective and there is a need to provide a much cheaper but reliable heat insulating product.

ZA-A-9701233 (corresponding to WO-A-9730951) describes the production of a non-combustible moulded part which comprises blowing vermiculite granules at elevated
20 temperature and then mixing with a binder. In that case, the binder must be phosphorus containing which is undesirable for applications with which the invention is concerned.

In accordance with a first aspect of the present
25 invention, a heat resistant product comprises a ceramic binder and vermiculite, wherein between 35% and 95% of the dry weight of the product is vermiculite having a particle size such that more than 60% of the vermiculite does not pass through a 1mm sieve.

30 In accordance with a second aspect of the present invention, a method of manufacturing a heat resistant product comprises mixing vermiculite granules with a ceramic binder; and drying the mixture, wherein between 35% and 95% of the dry weight of the product is vermiculite
35 having a particle size such that more than 60% of the vermiculite does not pass through a 1mm sieve.



We have found that a very cost effective product can be obtained by utilizing a mixture of vermiculite granules and a ceramic binder. This should be contrasted with known mica sheet and panels made using paper making or other processes which are very dense products. The new product will be much less dense than this sheet material with the result that heat insulation is promoted by virtue of the trapped air. Furthermore, the resultant product is very lightweight making it much easier to handle and can be utilized in a variety of forms depending upon the application.

We have realised that a problem with the known product is that it is made using ground or sieved particles which result in too high a density. This results in a reduction in voids which include trapped air and thus a reduction in the heat resistant performance of the product. On the other hand, if the particle size is too large then a product can result in which there is an open, air path from one side of the product to the other through which heat can be conducted. By utilising particles which fall within the inventive ranges, both these problems are overcome while at the same time a relatively light weight product is produced.

We have also found that the resultant product has good mechanical strength properties and for example with densities in the range $250\text{--}300\text{Kg/m}^3$, flexural strengths of $0.15\text{--}0.45\text{ MPa}$ are achieved. Experiments have shown that the density is preferably no less than 140Kg/m^3 to achieve acceptable flexural strength in a self-supporting product. Furthermore, the greater the proportion of the vermiculite, the more light weight the product. Thus, we have found that a product incorporating 95% dry weight vermiculite leads to a density of about 730Kg/m^3 while 35% dry weight vermiculite leads to a density of about 350Kg/m^3 .

Preferably, 50%-90% of the dry weight of the product is vermiculite having a particle size such that more than 60% of the vermiculite does not pass through a 1mm sieve.

In one form, the product is substantially rigid and can be handled rather like wood or the like so that it can be cut to required shapes. The product is relatively brittle and so typically will be sandwiched between load supporting sheets which are adhered to the product. These load supporting sheets can comprise fibre reinforced material sheets, steel or any other material enabling the panel to function as a structural or semi-structural component.

In other applications, the product is adhered to the surface of an article. Thus, a substantially rigid product could be produced which is then adhered to the article or more conveniently the product is formed in-situ on the article, for example by moulding, or by spraying onto the article.

An important application for the product is with known sandwich structures. These typically comprise of a core material such as foamed metals, plastics, wood, endgrain balsa or a cellular honeycomb of metal, plastic or composite materials sandwiched between planar members such as phenolic glass laminates or any other suitable material. These sandwich structures have considerable importance within structural engineering and are used extensively in aerospace and other industries since they provide key benefits over conventional materials including very low weight, high stiffness, durability and production cost savings. However, such sandwich structures do not generally operate above 170°C. If a product according to the invention is adhered to the surface, the structure can be used at higher temperatures. If the product is included as the core, the thermal conductivity and fire resistance is improved.

The product can be produced in a number of different thicknesses and consistency and can adhere to most surfaces including metal, wood and composites. The product can be moulded around complicated shapes and can be produced in very thin layers which are flexible. Glass fibre, woven

glass fibre or other fibrous materials can be used to reinforce the product if required. This is dependent on the thickness of the product and the binder used.

In principle, any ceramic binder could be used, such
5 a binder typically comprising an alkali silicate or blend with a filler such as alumina, magnesia, silica or other ceramic. The preferred binder comprises the adhesive part of a two part binder such as a blend based on sodium silicate. Commercial examples of such a binder are Al/CS
10 manufactured by Fortafix Limited, and Aremco 571. The Al/CS binder comprises a first part of a sodium silicate blend and a second part of an alumina filler. We have found that a very good product is obtained by using the adhesive part only (without the filler) or possibly by
15 replacing the filler with powdered vermiculite.

The step of drying the mixture could be carried out by heating or any other drying method including vacuum drying, optionally assisted by the use of microwave radiation.

The amount of vermiculite used will depend upon the
20 desired density and strength of the product and the manner in which it is used. If the product is supported between sheets then up to 95% of the dry weight of the product may be vermiculite. Lower proportions would be more suitable in other applications, a preferred range being 50%-90%.

25 The product can be made in a variety of ways and in the preferred approach, a two step method is carried out, step 1 comprising coating the particles with a ceramic binder, and curing/drying the binder. Step 2 may comprise coating the precoated particles with a ceramic binder
30 (possibly different from that used in step 1) and curing/drying the binder with or without pressure.

The drying step is conveniently carried out by heating the mixture, typically in the range 40-120°C. However,
35 other forms of drying such as vacuum drying which do not require the generation of heat could also be used. Typically, the mixture will be stirred to optimise the coating process. Alternatively, no stirring could be used

which will mean that the particles and binder will generate a consolidated mass which can then be broken up to generate individual, coated particles.

5 As with step 1, the drying part of step 2 could be carried out by heating the mixture or by some other method which does not involve heat such as vacuum drying.

The resultant product will be heat resistant and so can be used for firewalls and the like that is preferably also heat insulating.

10 Examples of applications of the invention are set out below:

1. Fire walls and doors for buildings and ships.
2. Protecting surfaces that are near to a heat source i.e. exhaust manifolds on cars and trucks.
- 15 3. Cable industry where once asbestos materials were used.
4. Electronic motors, commutators, insulators.
5. Cabin and cargo holds for aircraft.
- 20 6. Ceilings, walls and internal panels of trains.
7. Heat shields on high performance aircraft i.e. leading edges of wings, noses, and cones.
8. Protection from solar or other radiant heating.

Tests have shown that the heat insulating product according to the invention has a number of important properties:

1. Thermal properties, the product can withstand heat up to temperature of 1000°C.
2. Resists fire without burning.
- 30 3. Low heat conductivity which is density dependent.
4. Insulating electrical properties better than 24kV/mm.
5. Permeability to microwaves.
- 35 6. Good resistance to arcing and arc erosion.
7. Vermiculite is inert to most chemicals.

8. Mechanical properties are very good in particular compression resistance.

9. Good tensile and bending strength.

5 These properties together with density and mechanical strength, can be varied depending upon the particular mixture of vermiculite and binder and processing method which is used and these can be chosen depending upon the application.

10 Some examples of heat resistant products and methods for their manufacture according to the invention will now be described with reference to the accompanying drawings, in which:-

15 Figure 1 is a schematic cross-section through one example of a product incorporated onto a honeycomb structure;

Figure 2 illustrates the results of a fire test on the structure shown in Figure 1;

Figure 3 is a photograph of another example of the product; and,

20 Figure 4 illustrates graphically the relationship between flexural strength and density for a sample having a thickness of 25mm, a span length of 120mm and a loading rate of 2mm/min.

25 Figure 1 illustrates a honeycomb core 1 made, for example, of aluminium in a conventional manner. This core is sandwiched between a pair of phenolic skins 2,3 which are adhered to the core 1. As explained previously, this sandwich structure does not have significant fire resistance and so to improve its fire resistance, a vermiculite heat insulating layer 4 is provided on the phenolic skin 2. This was achieved in the following manner.

35 A conventional two part ceramic adhesive Al/CS was selected and 120 grams of the adhesive part (that is a blend of sodium silicate) was mixed with 2 grams of powdered vermiculite and 26 grams of vermiculite granules. The powdered vermiculite took the place of the conventional

alumina filler which is normally used with the ceramic adhesive and is used in the same proportion as the filler.

In this example, the granules comprised exfoliated vermiculite with particle sizes such that 93% of the
5 particles passed through an 8mm sieve, 32% through a 4mm sieve and 4% through a 2mm sieve.

The exposed surface of the phenolic skin 2 was degreased, this skin having dimensions of 150mm x 150mm x 12mm. 70 grams of an adhesive (Fortafix Al/CS)/powdered
10 vermiculite was applied onto the clean surface of the phenolic skin or board 2 and the board was placed in a bonding jig with the adhesive surface face up.

The mixture of vermiculite granules and adhesive was applied onto the precoated surface of the phenolic skin 2
15 to a thickness of about 8mm. A thin layer of powdered vermiculite was then evenly spread over the exposed surface of this previously coated mixture and the resultant product was clamped over its entire area using a steel caul plate with release paper. The structure was then placed in a
20 preheated oven for one hour at 60°C to bind the vermiculite granules into the layer 4 and adhere that layer to the phenolic skin 2. In some cases, further improved properties can be obtained by using higher temperatures.

It should be understood that the provision of a thin
25 layer of powdered vermiculite over the surface of the layer 4 is optional as is the use of powdered vermiculite in the earlier mixing process. It was found that the replacement of the powder filler or its omission from the binder provided a significant weight saving without adversely
30 effecting the performance as well as providing a cost saving.

The performance of the structure shown in Figure 1 was assessed by placing thermocouples onto the structure at the positions shown in Figure 1. A series 1 thermocouple 10
35 was placed on the surface of the phenolic skin 3, and a series 2 thermocouple 11 was placed between the phenolic skin 2 and the layer 4; and series 3 thermocouple was

placed on the uncoated, external surface of the layer 4. As can be seen from Figure 2, the fire test showed that when a flame at 1000°C was placed on the exposed surface of the layer 4, the series 1 thermocouple indicated that the temperature on the opposite surface of the structure did not exceed 130°C for 15 minutes. The series 2 thermocouple indicates that the temperature between the layer 4 and the phenolic skin 2 did not exceed 220°C.

In view of the significant reduction in heat across the layer 4, conventional adhesives can be used to adhere that structure to the support surface.

Figure 3 illustrates another form of the product which has a substantially rigid structure and is self-supporting. This can be used as the core between load bearing boards or plates, for example made of vermiculite (not shown). As can be seen in Figure 3, the product has substantial size and the granules are clearly visible, having a maximum dimension up to 15mm. The product can be handled in a similar way to wood as previously mentioned. This product has been formed by a moulding process and it will be clear that more complex shapes could also be achieved using appropriate moulds. This indicates the versatility of the invention.

The preferred method to produce a core material involves two stages.

The first stage requires vermiculite granules and a ceramic binder (in this example a blend of sodium silicate) to be mixed in a minimum ratio of 1:0.5 by weight of vermiculite to binder. This is then cured at a maximum of 120°C for a minimum of ¼ hour. This produces a precoated vermiculite for use in stage two.

Stage 2 requires precoated vermiculite from stage 1 to be coated with a ceramic binder in a minimum ratio of 1:0.5 by weight of precoated vermiculite to binder. This is then cured (usually in a mould) at a maximum temperature of 100°C for a minimum of ¼ hour, with the application of pressure.

This product is appropriate for use with or without adhered skins.

We have made some investigations into the variation of mechanical performance with density of samples of the product and the results are illustrated in Figure 4. As
5 can be seen, acceptable flexural strength is achieved at relatively low densities.

We have also performed a set of flexural tests on samples of the product, both with and without aluminium
10 skins. These tests were performed on samples of 250Kg/m³ density, 15mm thick, in accordance with the standard ASTM C-393, although a span length of 120mm was used.

Samples were tested with no skins, and with 1.5mm thick aluminium alloy skins. The results are presented
15 below:

	Average Flexural Strength (Mpa)
Without skins	0.31
With aluminium skins (1.5mm thick)	5.93

We have also investigated the thermal conductivity of
20 the product. This was done using a thermal imaging technique. The value obtained for a 250Kg/m³ density panel was 0.06W/m/K.

CLAIMS

1. A method of manufacturing a heat resistant product, the method comprising
- 5 (i) coating vermiculite granules with a ceramic binder, and curing/drying the binder to form precoated vermiculite granules; and
- (ii) coating the precoated vermiculite granules with a ceramic binder, and curing/drying the binder,

10 wherein between 35% and 95% of the dry weight of the product is vermiculite having a particle size such that more than 60% of the vermiculite does not pass through a 1mm sieve.

2. A method according to claim 1, wherein the curing/drying steps comprise heating or vacuum drying.

3. A method according to claim 1 or claim 2, wherein the mixture of precoated vermiculite granules and binder is held in a mould or press during curing/drying in step

15 (ii).

4. A method according to claim 1 or claim 2, wherein the mixture of precoated vermiculite granules and binder is coated onto a surface of an article prior to the curing/drying step.

20 5. A method according to any preceding claim, wherein 50%-90% of the dry weight of the product is vermiculite having a particle size such that more than 60% of the vermiculite does not pass through a 1mm sieve.

6. A method according to any preceding claim, wherein the vermiculite has a particle size such that more than 80% of the vermiculite does not pass through a 2mm

25 sieve.

7. A method accordingly to any preceding claim, wherein the binder comprises the adhesive part of a two part binder.

8. A method according any of claims 1 to 6, wherein the binder comprises the adhesive part of a two part binder, mixed with powdered vermiculite.

30 9. A method according to any preceding claim, wherein the vermiculite granules have a maximum dimension up to 15mm.

10. A heat resistant product obtainable by a method according to any preceding claim.

11. A product according to claim 10, wherein the product is substantially rigid.

12. A product according to claim 10 or claim 11, further comprising glass fibre or
5 other fibrous material reinforcement.

13. A product according to any of claims 10 to 12, which comprises voids which include trapped air.

14. A product according to any of claims 10 to 13, wherein the product is sandwiched between load supporting sheets adhered to the product.

10 15. A product according to any of claims 10 to 14 adhered onto the surface of an article.

16. A product according to any of claims 10 to 13 moulded onto the surface of an article.

15 17. A product according to any of claims 10 to 13 sprayed onto the surface of the article.

18. A product according to claim 16 or claim 17, wherein the article comprises a honeycomb structure.

19. A product according to claim 18, further comprising a phenolic glass laminate sandwiched between the honeycomb structure and the product.

20 20. A fire wall comprising a heat resistant product according to any of claims 10 to 19.