(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 245 360 A2

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
02.10.2002 Bulletin 2002/40

(51) Int Cl.[7]: **B28B 11/00**, C04B 38/00

(21) Application number: **02006786.4**

(22) Date of filing: **25.03.2002**

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE TR
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: **26.03.2001 JP 2001088345**
26.02.2002 JP 2002049695

(71) Applicant: **Denso Corporation**
Kariya-city, Aichi-pref., 448-8661 (JP)

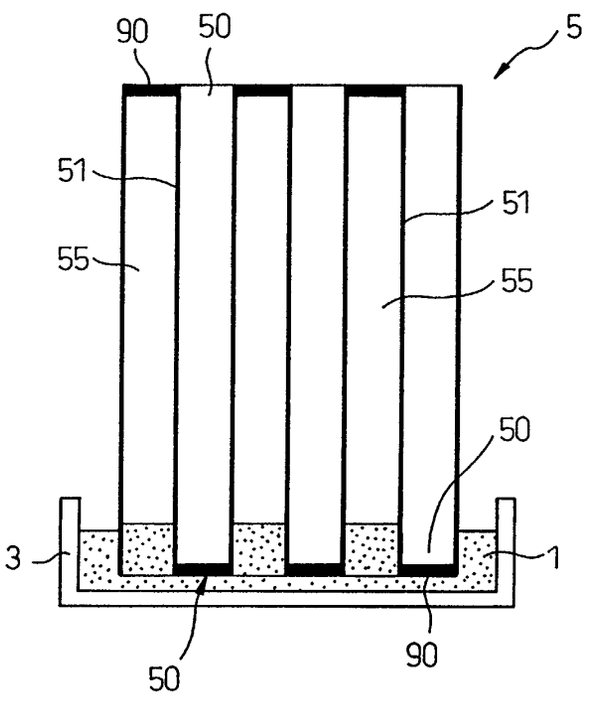
(72) Inventors:
- **Ishihara, Mikio**
  Kariya-city, Aichi-pref., 448-8661 (JP)
- **Nishimura, Mamoru**
  Kariya-city, Aichi-pref., 448-8661 (JP)

(74) Representative:
Leson, Thomas Johannes Alois, Dipl.-Ing.
Tiedtke-Bühling-Kinne & Partner GbR,
TBK-Patent,
Bavariaring 4
80336 München (DE)

(54) Plugging method for ceramic honeycomb body

(57) This invention provides a plugging method, for a ceramic honeycomb body, that does not create clearance in plug members closing cell end portions. To produce a ceramic honeycomb body 5 having a large number of cells 55 partitioned by partitions 51 and cell end portions 50 plugged by plug members, end faces of the ceramic honeycomb body are immersed in a plug member slurry 1 while the cell end portions 50 not provided with the plug member are covered with a masking material 90, and are then dried and heated. The plug member slurry 1 contains ceramic particles, a foaming material and water or an oil solvent.

Fig.1



EP 1 245 360 A2

Description

1. Field of the Invention

[0001] This invention relates to a plugging method for a ceramic honeycomb body for collecting Diesel particulates, for example.

2. Description of the Related Art

[0002] A filter structure used for collecting particulates in an exhaust gas of an automobile, for example, includes a ceramic honeycomb body 5 having a construction in which partition walls 51 partition a large number of cells 55 as shown in later-appearing Figs. 3(a) and 3(b), and which has a closing portion 15 where cell end portions 50 of some of the cells 55 are alternately closed by plug members 10.

[0003] To produce this ceramic honeycomb body 5, methyl cellulose as a binder, water and a stearic acid emulsion as a lubricant are mixed with silicon carbide powder to prepare a kneaded material, as described in Japanese Unexamined Patent Publication (Kokai) No. 9-25180. Extrusion molding of the kneaded material is carried out to obtain a ceramic honeycomb body having cell end portions 50 at both ends of each cell 55. Plug members 10 are put into some of the cell end portions opening at both end faces.

[0004] In this plugging operation, a masking material 90 such as wax is applied in a checkerwork pattern to every other cell end portions 50 of the ceramic honeycomb body 5 (see Fig. 3b). Both end portions of this ceramic honeycomb body 5 are immersed in the plug material slurry to let it soak into the cell end portions 50. The ceramic honeycomb body 5 is then dried and heated.

[0005] In consequence, the plug member 10 enters into each cell end portion of the ceramic honeycomb body 5 that is not masked by the masking material 90. The masking material 90 is thereafter removed. As a result, there is obtained the ceramic honeycomb body 5 having inlets 14 and outlets 16 alternately formed as shown in Figs. 3a and 3b.

[0006] However, the plugging method of the ceramic honeycomb body according to the prior art involves the following problem.

[0007] In the ceramic honeycomb body 5 described above, the plug member 10 closing each cell end portion 50 should fully close the cell end portion as shown in Fig. 6(a).

[0008] However, when the plug member slurry is charged into the cell end portions 50 and is thereafter dried and heated to form the plug members 10, the plug member slurry undergoes shrinkage.

[0009] In consequence, a clearance 109 develops in the plug member 10 closing each cell end portion in some of the cell end portions 50 as shown in Fig. 6(b).

[0010] If such a clearance 109 develops, an exhaust gas leaves the cell portion through the clearance when the exhaust gas is introduced into the cell portion of the ceramic honeycomb body 5. In this case, it is not possible to collect particulates in the exhaust gas.

SUMMARY OF THE INVENTION

[0011] In view of the problem of the prior art described above, this invention provides a plugging method of a ceramic honeycomb body that does not create a clearance in plug members for closing cell end portions.

[0012] According to a first aspect of the invention, there is provided a plugging method of cell end portions for producing a ceramic honeycomb body having a large number of cells partitioned by partitions and cell end portions plugged by plug members, by the steps of covering cell end portions not requiring the plug members with a masking material on the end faces of the ceramic honeycomb body, immersing the end faces of the ceramic honeycomb body into a plug member slurry and plugging the cell end portions, wherein the plug member slurry contains ceramic particles, a foaming material and water or an oil solvent.

[0013] In this invention, the plug member slurry contains the ceramic particles, the foaming material and water or the oil solvent. The plug member slurry is allowed to soak into the cell end portions not covered with the masking material, and the cell end portions are closed.

[0014] The foaming material in the plug member slurry at the cell end portions is foamed to thereby expand the plug member slurry (Fig. 4) and to form the plug member at each cell end portion.

[0015] Consequently, the plug member completely closes each cell end portion, and clearance is not created in the plug member.

[0016] According to a second aspect of the invention, there is provided a plugging method of cell end portions for producing a ceramic honeycomb body having a large number of cells partitioned by partitions and cell end portions plugged by plug members, by the steps of covering cell end portions not requiring the plug members with a masking material on end faces of the ceramic honeycomb body, immersing the end faces of the ceramic honeycomb body into

a plug member slurry and plugging the cell end portions, wherein the immersion operation into the plug member slurry is carried out a plurality of times.

[0017] Even when clearance occurs in the plug member when the cell end portion is immersed only once in the plug member slurry, this method can reliably prevent the occurrence of clearance in the plug member because the immersion operation is carried out a plurality of times.

[0018] Therefore, this invention can provide a plugging method of a ceramic honeycomb body that does not create a clearance in a plug member for closing each cell end portion.

BRIEF DESCRIPTION OF THE DRAWINGS

[0019]

Fig. 1 is an explanatory view showing a state where an end part of a ceramic honeycomb body is immersed in plug member slurry in Embodiment 1;

Fig. 2 is an explanatory view showing a state where an end part of the ceramic honeycomb body is immersed upside down in plug member slurry in Embodiment 1;

Figs. 3(a) is an explanatory sectional view of the ceramic honeycomb body in Embodiment 1, and Fig. 3(b) is an explanatory view of end portions of the ceramic honeycomb body the cell end portions of which are closed in a checkerwork pattern;

Fig. 4 is an explanatory view showing a swelling state of plug member slurry charged into the cell end portions in Embodiment 1;

Fig. 5 is a graph showing the relation between an addition amount of a foaming material and strength of a plug member in Embodiment 3; and

Fig. 6(a) shows a normal state of a plug member in a prior art example, and Fig. 6(b) is an explanatory view showing a state of the plug member where clearance develops.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0020] In the first or second aspect of the invention, the ceramic honeycomb body to be immersed in the plug member slurry may be one that has not been sintered. When the ceramic honeycomb body is not sintered, it is possible to produce the plug member described above by conducting sintering and foaming the foaming material. When the ceramic honeycomb body is sintered, it is possible to produce the plug member by foaming the foaming material in the plug member slurry at the cell end portions of the ceramic honeycomb body.

[0021] In the first aspect of the invention, a heat-foamable foaming material that undergoes foaming upon heating can be used as the foaming material described above.

[0022] It is preferred to use a material foaming at 80°C or above as the heat-foamable foaming material. An example of such a heat-foamable foaming material is foaming particles prepared by encapsulating a liquefied hydrocarbon type foaming agent in a thermoplastic resin.

[0023] The heat-foamable foaming material is prepared by encapsulating a liquid gas inside a polymer shell made of a thermoplastic resin. When this heat-foamable foaming material is heated, the gas pressure of the liquefied gas inside the polymer shell increases and at the same time, the polymer shell is softened. Consequently, its volume drastically increases and the foaming material results in hollow spherical particles.

[0024] Next, the foaming material is preferably contained in an amount of 0.1 to 5wt% on the basis of 100wt% of the ceramic particles.

[0025] In this case, the plug member can be more reliably packed into the cell end portions for closing them, and can prevent the occurrence of the clearance.

[0026] When the amount of the foaming material is less than 0.1wt%, the effect of the addition of the foaming material is small, in some cases. When the amount exceeds 5wt%, expansion of the plug member slurry becomes so great that compactness of the plug member is sometimes lost.

[0027] Next, the oil solvent is preferably an alcohol or a petroleum solvent.

[0028] In this case, the plugging operation can be made to a dried body before sintering, and the effect of reducing the cost can be obtained.

[0029] Next, the plug member slurry preferably contains 0.1 to 5wt% of the foaming material, 0.1 to 1wt% of the binder and 30 to 40wt% of water on the basis of 100wt% of the ceramic particles.

[0030] In this case, the plug member slurry can be more reliably packed into the cell end portion, and the occurrence of the clearance at the plug member can be prevented.

[0031] The reason for limitation of the addition amount of the foaming material is the same as described above.

[0032] When the binder content is less than 0.1wt%, it is difficult to fully pack the plug member slurry into the cell

end portions. When it exceeds 1%, on the other hand, the viscosity of the plug member slurry becomes so high that the plug member slurry cannot easily soak into the cell end portions.

[0033] When the water content is less than 30wt%, the viscosity of the plug member slurry becomes so low that the plug member slurry cannot be easily retained inside the cell end portion.

[0034] The ceramic particles contained in the plug member slurry are preferably the same kind as the ceramic honeycomb body or those which have a shrinkage ratio during sintering and a coefficient of thermal expansion approximate to those of the ceramic honeycomb body. Such ceramic particles can prevent the occurrence of cracks.

[0035] Examples of the ceramic particles are cordierite, a cordierite formation material prepared by mixing talc, kaolin, alumina, etc, in a predetermined ratio, mullite, silicon carbide and silicon nitride.

[0036] The binder described above plays the role of bonding the ceramic particles to one another. Examples of such a binder are celluloses such as methyl cellulose and ethyl cellulose, acrylic type binders and polyvinyl alcohol. The binder can be omitted depending on the combination of the kind of the ceramic particles and the adjuvant inside the plug member slurry.

[0037] The particle size of the foaming material is preferably from 5 to 30 μm (claim 5). When the particle size is less than 5 μm, the expansion ratio of foaming becomes small and the effect becomes small, too. When the particle size of the foaming material exceeds 30 μm, each pore becomes so large that clearance is likely to develop.

[0038] In the first and second aspect of the invention, plugging of the cell end portions is preferably carried out by the steps of immersing the end face of the ceramic honeycomb body into the plug member slurry and then conducting drying and heating.

[0039] When drying and heating are carried out as described above, the foaming material in the plug member slurry is allowed to foam at each cell end portion to form the plug member at the cell end portion.

[0040] After drying and heating of the honeycomb molded article are carried out as described above before sintering, the honeycomb molded article is sintered, so that the foaming material is allowed to foam and the plug member can be formed.

[0041] Next, preferred embodiments of the invention will be explained.

Embodiment 1:

[0042] A plugging method of a ceramic honeycomb body according to this embodiment will be explained with reference to Figs. 1, 2, 3(a), 3(b) and 4.

[0043] To produce a ceramic honeycomb body 5 having a large number of cells 55 partitioned by partitions 51 and plugged at cell end portions 50 by plug members 10 as shown in Figs. 1, 2, 3(a) and 3(b), the plugging method of this embodiment is carried out by the steps of covering each cell end portion 50, at which the plug member 10 is not required, with a masking material 90, immersing the end face of the ceramic honeycomb body 5 into a plug member slurry 1 and then conducting drying and heating.

[0044] The plug member slurry 1 contains ceramic particles, a foaming material, water or an alcohol and an oil solvent such as petroleum.

[0045] An explanation will be given in detail.

[0046] The plug member slurry in this embodiment is prepared by mixing 18wt% of fused silica (SiO_2) as the ceramic particles, 38wt% of talc ($MgO \cdot SiO_2$) and 44wt% of aluminum hydroxide, adding 2wt% of the foaming material and 35wt% of water to 100wt% of these ceramic particles, and mixing them together.

[0047] The foaming material is heat-foamable particles that undergoes foaming upon heating. These particles are formed by coating foaming material of a liquid hydrocarbon with a thermoplastic resin, and the particle size is about 15 μm.

[0048] The ceramic particles described above have a diameter of 1 to 40 μm.

[0049] To produce the ceramic honeycomb body 5, on the other hand, a mixture of the ceramic particles and the binder is extrusion-molded into a honeycomb shape and is sintered. The masking material is arranged at every other cell end portions 50 in a checkerwork pattern to close the cell end portions 50. The masking material 90 uses a wax. In this way, there is prepared the ceramic honeycomb body 5 in which the cell end portions 50 of the adjacent cells 55 are alternately closed by the masking material 90.

[0050] To close each cell end portions by the plug member 10, the plug member slurry 1 is placed into a vessel 3 as shown in Fig. 1 and one of the ends of the ceramic honeycomb body 5 is immersed into the plug member slurry 1. Consequently, the plug member slurry 1 soaks into each cell end portion 50 not closed by the masking material 90.

[0051] After the immersion operation is conducted as described above, the ceramic honeycomb body 5 is pulled out from the plug member slurry 1, and the plug member slurry is dried at about 100°C for 10 minutes.

[0052] Next, the ceramic honeycomb body 5 is turned upside down and the other end portion is immersed into the plug member slurry 1, is pulled out and is dried, as shown in Fig. 2.

[0053] Consequently, there is obtained a ceramic honeycomb body 5 in which the plug member slurry 1 soaks into

and fills the cell end portions 50 not closed by the masking material 90 as shown in Fig. 2.

[0054] Next, the ceramic honeycomb body 5 is placed into a heater and is gradually heated to about 1,400 to 1,450°C so as to sinter the ceramic particles 1 in the plug member slurry and to form the plug member 10. As a result, there is obtained the ceramic honeycomb body 5 having the closed portions 15 where the plug members 10 obtained from the plug member slurry 1 close the cell end portions 50 (Figs. 3(a) and 3(b)).

[0055] During heating described above, the wax of the masking material 90 is evaporated and removed, and the cell end portions 50 are opened.

[0056] In the resulting ceramic honeycomb body 9, the plug members 10 close every other cell end portions in the checker work as shown in Fig. 3(b). The intrusion length of the plug member 10 is about 4 mm.

[0057] Each partition 51 of the ceramic honeycomb body 5 is porous. Therefore, as indicated by arrows in Fig. 3(a), an exhaust gas entering from an inlet 14 of the ceramic honeycomb body 5 flows through the cell 55, passes through the partition 51, enters an adjacent cell 5 having an outlet 16 and is discharged from the outlet 16. In the meantime, the partition 51 collects Diesel particulates.

[0058] It is important in this embodiment that the plug member slurry 1 contains the ceramic particles, the foaming material, water or the alcohol and the oil solvent such as petroleum. The plug member slurry 1 impregnates the cell end portions 50 and is then dried. When the ceramic particles are heated for sintering, the ceramic particles are sintered with the partitions of the ceramic honeycomb body and close the cell end portions 50.

[0059] The foaming material mixed with the ceramic particles is foamed at this time by heating and the plug member slurry 1 is expanded. Sintering proceeds under this state, thereby giving the plug member. Therefore, the plug member 10 completely closes the cell end portions 50 without forming a clearance in the plug member.

[0060] Fig. 4 shows the state where the plug member slurry 1 is heated and expanded. As can be seen from this drawing, the foaming material in the plug member slurry 1 expands during heating, and a pushing force (indicated by arrows) operates outward. Therefore, the clearance does not develop in the plug member 10, and the ceramic particles and the plug member 10 come into close contact with the partitions 51. Therefore, the sintering strength is improved between the plug member 10 and the partition 51.

Embodiment 2:

[0061] This embodiment conducts the immersion operation described above three times without the foaming material used in Embodiment 1 being added.

[0062] The immersion operation is carried out while a drying step at about 80°C is interposed between them. As the immersion operation is conducted three times in this embodiment, the plug member slurry 1 can sufficiently soak into the cell end portions 50, and the plug member 10 can be formed without creating a clearance. Other effects are the same as those of Embodiment 1.

Embodiment 3:

[0063] In this embodiment, the optimal amount of addition of the foaming material in Embodiment 1 described above is confirmed through experiment. In the embodiment, the plug member 10 is formed at each cell end portion 50 of each ceramic honeycomb body 5 when the amount of addition of the foaming agent is changed, and the strength of the plug member 10 is measured. Whether or not any defect, such as clearance, in this plug member 10 exists is also determined.

[0064] The plug member slurry 1 in this embodiment contains the ceramic particles, the foaming material and "dry solvent" (a product of Omoteya Sekiyu K. K.).

[0065] With the exception of this point, the plug member 10 is produced in the same way as the plugging method of the ceramic honeycomb body 5 of Embodiment 1.

[0066] In this embodiment, the amount of addition of the foaming material on the basis of 100wt% of the ceramic particles is changed between 0 and 20wt% to form the plug member 10. The strength of this plug member 10 is measured, and whether or not a defect, such as clearance, exists in this plug member 10 is determined.

[0067] Fig. 5 shows the relation between the amount of addition of the foaming material and the strength of the plug member 10 so measured.

[0068] It can be appreciated from Fig. 5 that the greater the amount of addition of the foaming material, the lower becomes the strength of the plug member 10. It can also be appreciated that when the amount of addition of the foaming agent exceeds 5wt%, the strength of the plug member 10 remains constant but at a low level.

[0069] Table 1 represents the relation between the amount of addition of the foaming material and the number of defects of the plug members 10 inspected. The number of defects in this table represents the number of the plug members 10 in which clearance is found among the plug members 10 at a large number of cell end portions 50. (The ceramic honeycomb body 5 in this ceramic honeycomb body 5 has about 12,000 cell end portions on both end faces, and about 6,000 plug members 10 are produced).

Table 1

Addition amount of foaming material (wt%)	Number of defects
0	43
0.1	7
0.5	0
1	0
5	8
10	≧ 100
20	≧ 100

[0070] It can be understood that when the amount of addition of the foaming material does not exist at all (that is, 0wt%), the number of defects is greater and when the amount of addition of the foaming material is greater than 10wt%, too, the number of defects is great.

[0071] Therefore, it can be understood from the measurement of the strength and from the inspection of the number of defects that the optimal amount of addition of the foaming agent is 0.1 to 5wt%.

[0072] This invention provides a plugging method, for a ceramic honeycomb body, that does not create clearance in plug members closing cell end portions. To produce a ceramic honeycomb body 5 having a large number of cells 55 partitioned by partitions 51 and cell end portions 50 plugged by plug members, end faces of the ceramic honeycomb body are immersed in a plug member slurry 1 while the cell end portions 50 not provided with the plug member are covered with a masking material 90, and are then dried and heated. The plug member slurry 1 contains ceramic particles, a foaming material and water or an oil solvent.

Claims

1. A plugging method, for cell end portions, for producing a ceramic honeycomb body having a large number of cells partitioned by partitions and cell end portions plugged by plug members by the steps of covering cell end portions not requiring said plug members with a masking material on end faces of said ceramic honeycomb body, immersing the end faces of said ceramic honeycomb body into a plug member slurry and plugging said cell end portions, wherein said plug member slurry contains ceramic particles, a foaming material and water or an oil solvent.

2. A plugging method for a ceramic honeycomb body according to claim 1, wherein said foaming material is contained in an amount of 0.1 to 5wt% on the basis of 100wt% of said ceramic particles.

3. A plugging method for a ceramic honeycomb body according to claim 1, wherein said oil solvent is a petroleum solvent.

4. A plugging method for a ceramic honeycomb body according to claim 1, wherein said plug member slurry contains 0.1 to 5wt% of said foaming material, 0.1 to 1wt% of a binder and 30 to 40wt% of water on the basis of 100wt% of said ceramic particles.

5. A plugging method for a ceramic honeycomb body according to claim 1, wherein the diameters of the particles said foaming material is 5 to 30 μm.

6. A plugging method for a ceramic honeycomb body according to claim 1, wherein said cell end portions of said honeycomb molded article are plugged by the steps of immersing said end faces into said plug member slurry and then conducting drying and heating.

7. A plugging method for cell end portions, for producing a ceramic honeycomb body having a large number of cells partitioned by partitions and cell end portions plugged by plug members by the steps of covering cell end portions not requiring said plug members with a masking material on the end faces of said ceramic honeycomb body, immersing the end faces of said ceramic honeycomb body into a plug member slurry and plugging said cell end portions, wherein said immersion into said plug member slurry is carried out a plurality of times.

8. A plugging method for a ceramic honeycomb body according to claim 6, wherein said cell end portions of said honeycomb molded article are plugged by the steps of immersing said end faces into said plug member slurry and then conducting drying and heating.

Fig.1

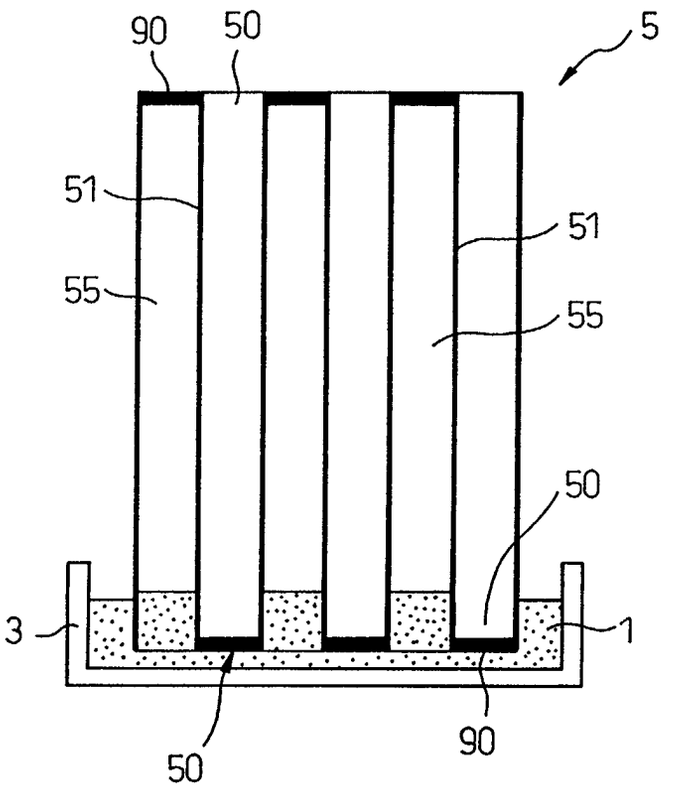


Fig.2

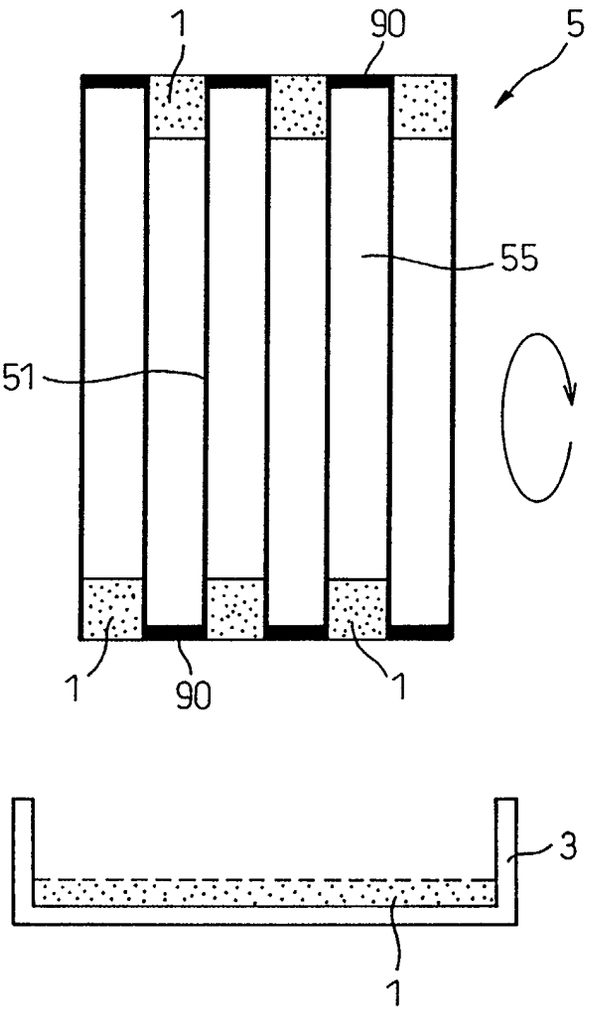


Fig.3(a)

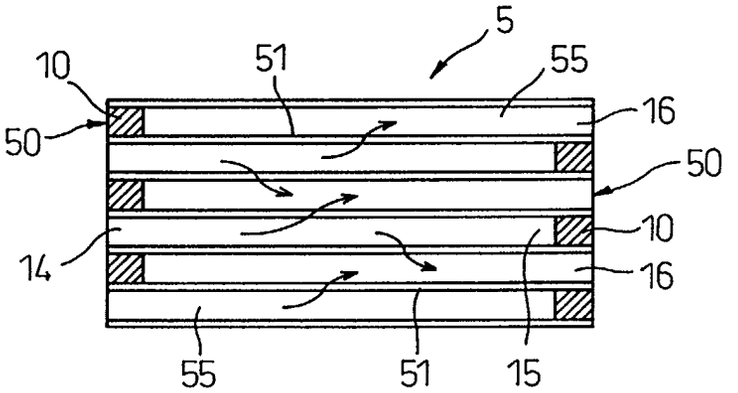


Fig.3(b)

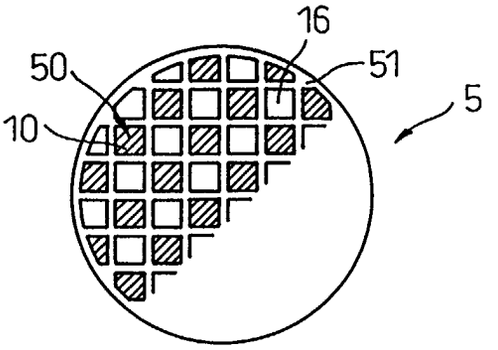


Fig.4

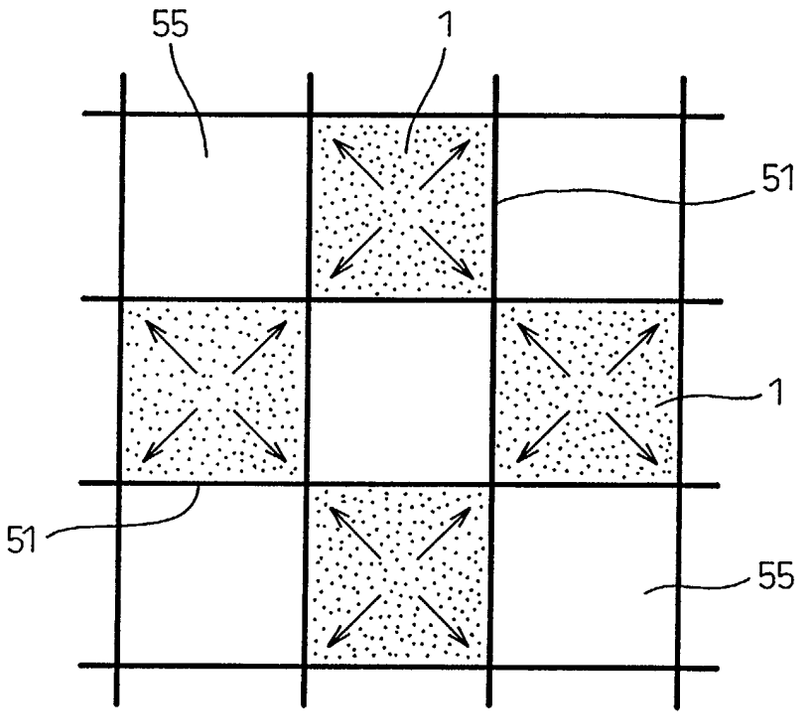


Fig.5

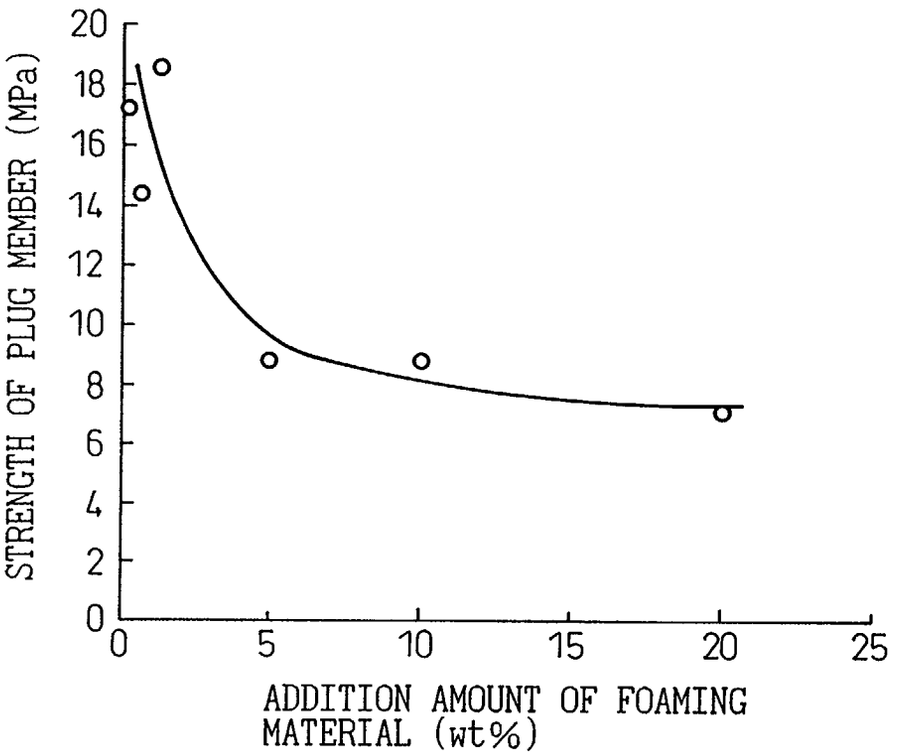

STRENGTH OF PLUG MEMBER (MPa)
20
18
16
14
12
10
8
6
4
2
0
0
5
10
15
20
25
ADDITION AMOUNT OF FOAMING MATERIAL (wt%)

Fig.6(a)

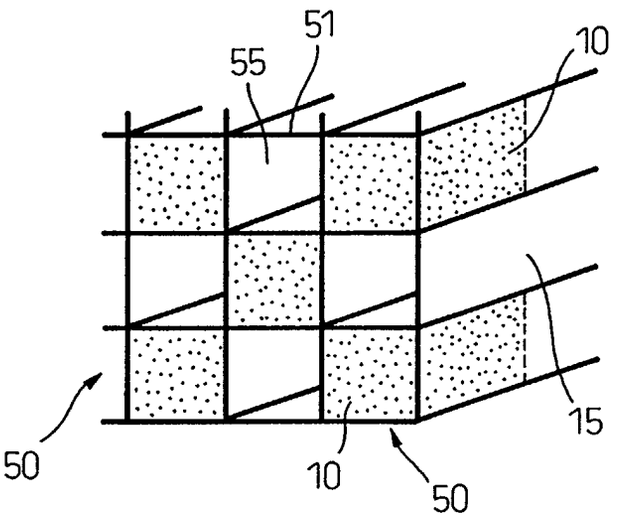


Fig.6(b)