



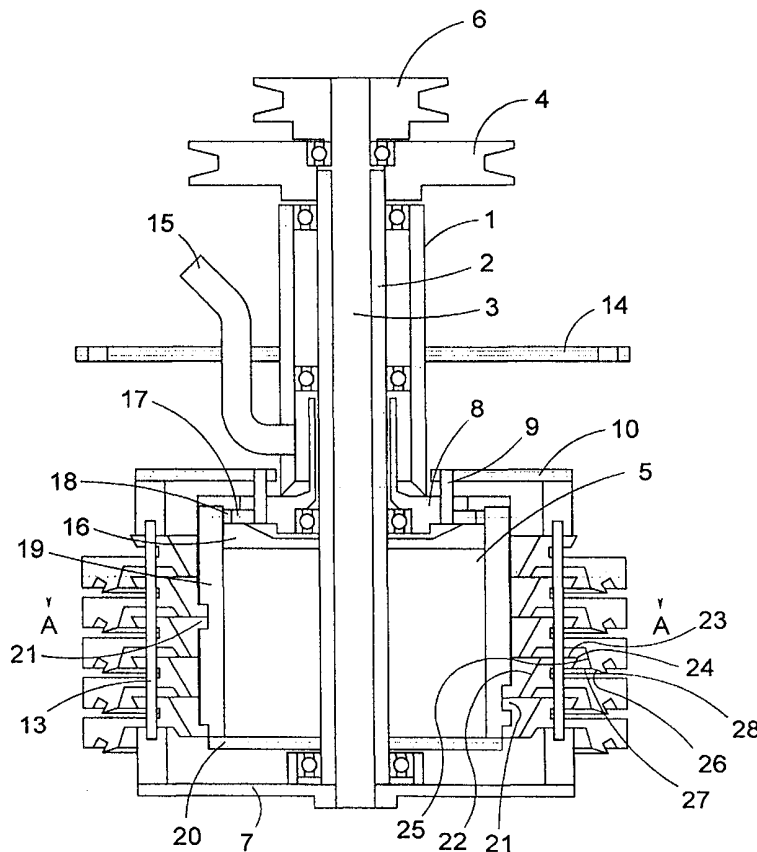
INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification ⁷ : B22F 1/00, 9/10, B01J 2/14, B05B 3/02</p>	<p>A1</p>	<p>(11) International Publication Number: WO 00/18529</p> <p>(43) International Publication Date: 6 April 2000 (06.04.00)</p>
<p>(21) International Application Number: PCT/SE99/01698</p> <p>(22) International Filing Date: 24 September 1999 (24.09.99)</p> <p>(30) Priority Data: 9803289-9 25 September 1998 (25.09.98) SE</p> <p>(71) Applicant (for all designated States except US): SANDVIK AB (publ) [SE/SE]; S-811 81 Sandviken (SE).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): ANDERSSON, Alf [SE/SE]; Box 23, S-260 35 Ödåkra (SE). NELANDER, Bo [SE/SE]; Ärjavägen 14, S-125 41 Älvsjö (SE). JUTTERSTRÖM, Ulf [SE/SE]; Lidners plan 11, S-112 53 Stockholm (SE).</p> <p>(74) Agent: TÅQUIST, Lennart; Sandvik AB, Patent Dept., S-811 81 Sandviken (SE).</p>	<p>(81) Designated States: IL, JP, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</p> <p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments. In English translation (filed in Swedish).</i></p>	

(54) Title: METHOD AND MEANS FOR DRYING CEMENTED CARBIDE AND SIMILAR

(57) Abstract

The invention teaches a new method and an apparatus for manufacturing powders of cemented carbide, cermets, ceramics and similar materials with good abrasive wear resistance starting from a slurry whereby said slurry is introduced into a drop forming device including at least one essentially horizontal oriented rotating drop forming disk (12), from which the drops are slung out to solidify by means of the centrifugal force. According to the invention the slurry is ejected from a discharge opening (21) on the drop former in the form of a jet such that it hits a first obliquely downwards-inwards directed surface (22) whereby the speed of rotation of the disk is chosen such that the jet of liquid by the centrifugal force after that is forced upwards over the first surface as well as over to another connected horizontal surface (23) and thereafter is ejected as a split jet against a second obliquely downwards-outwards directed surface (25) such that the slurry is accelerated to the speed of the drop forming disk (12) and then is led over a third surface (26) obliquely directed outwards, from where the drops are then caused to detach themselves and fall down forming a powder.



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METHOD AND MEANS FOR DRYING CEMENTED CARBIDE AND SIMILAR

The present invention relates to a method for drying powder mixtures of cemented carbide and the like.

5 Cemented carbide is made by powder metallurgical methods consisting of the wet milling in an alcohol-water-solution of a powder mixture containing powder forming the hard constituents and a binder phase, drying of the milled mixture to a powder with good flow properties by spray drying, pressing of the dried powder to bodies of desired shape and finally sintering.

10 The intensive milling operation is performed in mills of different sizes using cemented carbide milling bodies. Milling is considered necessary to obtain a uniform distribution of the binder phase in the milled mixture. The milling time can vary from several hours up to days. The milling operation produces a slurry suitable for spray drying. Successful spray drying depends strongly on the properties of the slurry. The
15 present technology with intensive milling under long periods of time usually gives a very fine-grained powder whose sedimentation rate is sufficiently slow. As result of the spray drying a ready-to-press powder is obtained consisting essentially of spherical agglomerates of about 0.1 mm mean size.

It is desirable that the spray-dried powder shall have spherical well-defined
20 agglomerates with a limited variation of the sizes of the agglomerates. In such a way that a more even powder density is obtained, giving improved flow properties as well as a reduction of filling variations in the subsequent pressing operation. The distribution can be influenced by optimising the alcohol to water ratio and liquid to powder ratio in the slurry, the amount of pressing agent etc. Another way is to modify
25 the nozzles in the spray drier. A variant of such a nozzle is described in US 4,978,069. This patent relates to the generation of even drops from a melt but it can also be applied to drying of cemented carbide powder. However, it has been found that this does not give the desired limited variation of the sizes of the agglomerates. It has been found better to use a simpler nozzle instead and expose the powder to a fractioned
30 sieving after the drying process to eliminate the coarse respectively fine part of the agglomerates to obtain the desired distribution of sizes. The distribution after the sieving must be controlled since there is a risk of clogging of the net of the sieve, which may influence the results. The sieving also means that the powder is affected so that poorer properties are obtained. However, the distribution of sizes of the
35 agglomerates is normally so broad that the final distribution cannot be made as narrow as desirable for economical reasons. A way to describe distribution of the sizes of the agglomerates is through the expression d_{97}/d_{03} where d_{97} = the grain size below which 97 % of the agglomerates are found and d_{03} = the grain size below which 3 %

of the agglomerates are found. A d_{97}/d_{03} value = 4 has been found to be acceptable. It is, however, desirable to obtain an even lower value preferably without a subsequent sieving operation.

It is an object of the present invention to provide a method for the production
5 of cemented carbide powder with a limited distribution of grain sizes $d_{97}/d_{03} < 4$ without subsequent sieving.

Surprisingly it has now been found that if the apparatus, according to the above mentioned US-patent, is modified in the way more closely described below a cemented carbide powder is obtained with the desired limited distribution of grain
10 sizes.

In an apparatus for drop forming by disks, according to the present invention, jets of the liquid are ejected towards a first funnel-shaped part where the liquid is accelerated in a first step after which the jets are permitted to spread somewhat, they are then ejected over to a second funnel-shaped part where the liquid is accelerated in
15 a second step to the speed of the drop forming disk. The acceleration of the liquid is thus divided into two steps. During the second acceleration step the acceleration is increased because the spreading of the jet results in increased surface contact.

At the periphery of the drop forming disk the risk that contact between the surface of the disk and the liquid be broken is minimised thanks to the fact that the
20 outermost part of the disk is angled relative to the plane of rotation in such a way the centrifugal force acting on the liquid is split into one component directed along the surface of the disk and one directed against the surface of the disk. In this way the contact between the surface of the disk and the layer of liquid right up to the means of drop forming is ensured.

25 Fig 1 shows the drop forming means in a longitudinal section view.

Fig 2 shows a section along the line A-A in Fig 1.

Fig 3 shows part of a drop-forming disk in greater detail.

Fig 4 shows agglomerates dried according to known technique in a 30X magnification.

30 Fig 5 shows agglomerates dried according to the invention in a 30X magnification.

More particularly the means of drop forming according to the invention consists of a bearing housing 1 in which a tube shaped shaft 2 is mounted, in which a shaft 3 is mounted. A belt pulley 4 and a slinger rotor 5 are mounted on the shaft 2,
35 and a belt pulley and a lower disk 7 are mounted on the shaft 3. A distributor disk 8, to which is connected an upper disk by means of rods 13 is mounted on the shaft 2. The bearing housing 1 has a flange 14 for the mounting of the drop forming apparatus in e.g. a drying chamber and an in-feed tube 15 for the liquid. The belt pulley 6, the

shaft 3, lower disk 7, the slinger rotor 5, the upper disk 10 and the distributor disk 8 constitute a rigidly inter-connected rotatable unit. The slinger rotor 5 has a receiving disk 16 with a groove 17 in the bottom of which are formed uniformly distributed, even sized holes 18, to which are arranged axially proceeding channels 19, which are
5 connected at their lower ends to a disk 20. The channels 19 have discharge openings 21 arranged at the funnel-shaped part 22 of the drop forming disks. This part transforms into an essentially plane part 23 with a sharp edge 24, directed towards a second funnel-shaped part 25, which in its lower part transforms to a surface 26, which is directed at an angle 27 towards the plane of rotation of the drop forming
10 disk. The surface 26 extends radially to a sharp edge 28, on which (not shown) are arranged means of drop forming in the shape of uniformly distributed, uniform, protruding bumps.

An arrangement according to the invention has suitably the following dimensions:

15 Radius of the distributor disk 8: 12.5 – 20 cm.

Radius of the lower disk 7: 10-15 cm.

Number of drop forming disks 5: 10-25.

Angle of the funnel-shaped part 22: 45-55°, preferably about 50° to the horizontal plane.

20 Angle of the funnel-shaped part 25: 65-75°, preferably about 70° to the horizontal plane.

Angle of the funnel-shaped part 26: <10°, preferably about 5° to the horizontal plane.

25 With these dimensions a drying capacity of 100 – 500 kg cemented carbide powder/hour is obtained.

When the drop forming arrangement is used for drop forming the two belt pulleys 4, 6 and thereby also the shafts 2, 3 and the means connected thereto are rotated. Liquid is supplied through the supply tube 15 and flows down onto the distributing disk 8, from which it is ejected against the groove 17. The shaft 3 is
30 generally rotated with considerably higher speed than the shaft 2 which results in the liquid being ejected from the distributor disk 8 into the groove 17 and it is then brought to flow into the groove 17 in the direction of rotation of the distributor disk. The liquid which hits the groove 17 in a part which is limited by the farther edge of a hole 18 and that of the next hole 18 seen in the direction of the flow of the liquid and
35 it flows out via the farther hole 18 and is led further away via the channel 19. The accuracy of the distribution is further improved in that each and every one of the holes 18 continuously occupies all the positions on the circumference. The liquid is ejected from the discharge opening 21 in the form of a jet towards the funnel-shaped part 22

of the drop-forming disk 12. The liquid is accelerated in the direction of the motion of the drop forming disk and moves in connection herewith outwards/upwards over the funnel-shaped part 22 and the plane part 23 and is ejected as a divided jet towards the other funnel shaped part 25, where the liquid is accelerated to the speed of the drop forming disk 12 and led out over the surface 26 to the means of drop forming from which the drops are released.

EXAMPLE 1

A cemented carbide slurry with the composition 6% Co and the balance WC was dried using a drop generator according to the invention and was compared to material with corresponding composition which had been spray dried with a pressure nozzle and subsequently sieved (known technique). The generator according to the invention consisted of twelve disks and it had a diameter of 330 mm. The drying capacity was ca 450 kg/h.

After drying distribution of sizes in the agglomerate was determined by sieve analysis as well as laser diffraction analysis. It was found, surprisingly, that a generator according to the invention gave such a narrow distribution of sizes and that a distribution factor of $d_{97}/d_{03} = 3.6$ could be obtained essentially without sieving. Powder dried with nozzle required for a distribution factor of $d_{97}/d_{03} = 4$ extraction by sieving of 15 weight-% powder and a distribution factor of $d_{97}/d_{03} = 3$ extraction by sieving of more than 20 weight-%.

The dried powders were also studied in a microscope and it was found that powder dried according to invention exhibited not only more even agglomerate size distribution but also a better quality of the agglomerates, see Fig 4 and 5.

Flow measurement according to Hall Flow ISO 4490 for powder dried according to invention gave a value of 31-32 s and for known technique 34-35 s.

Claims

1. Method of making powders of cemented carbide, cermets, ceramics and similar materials with good abrasive wear resistance starting from a slurry whereby said slurry is introduced into a drop forming apparatus including at least one
5 essentially horizontally oriented, rotating, drop forming disk (12), from which the drops by means of the centrifugal force are slung out to solidify, whereby the slurry in said arrangement is uniformly distributed circumferentially relative to a vertical axis of the said around the axis rotating drop forming disk c h a r a c t e r i s e d by the fact that the slurry is ejected out from a discharge opening (21) of the drop former in the
10 shape of a jet such that it hits a first obliquely downwards-inwards directed surface (22) whereby the speed of rotation of the disk is chosen such that the jet of liquid is forced upwards over the first surface as well as over to another connected horizontal surface (23) by the centrifugal force and thereafter is ejected as a divided jet towards a second obliquely downwards-outwards directed surface (25) such that the slurry is
15 accelerated to the speed of the drop forming disk (12) and is then led over a third surface (26) obliquely directed outwards, from where the drops are then caused to detach themselves and fall down forming a powder when the centrifugal force exceeds the adhesive force which retains the drops.

2. Method according to claim 1 c h a r a c t e r i s e d in that the first obliquely
20 downwards-inwards sloping surface (22) as well as the horizontal surface (23) connected thereto are arranged as integrated parts of one and the same disk whereas the second oblique downwards-outwards directed surface (25) is arranged as a surface of an axially mounted disk shaped section on top of the first drop disk.

3. Method according to claim 1 or 2 c h a r a c t e r i s e d in that the first
25 surface (22) is arranged as a surface with an inclination angle of 45-55° relative to the horizontal plane.

4. Method according to any of claims 1-3 c h a r a c t e r i s e d in that the second surface (25) is arranged as a surface with an inclination angle of 65-75° relative to the horizontal plane.

30 5. Method according to any of claims 1-4 c h a r a c t e r i s e d in that the third obliquely outwardly directed surface (26) is arranged as a surface with an inclination angle of <10° relative to the horizontal plane.

6. Apparatus for the breaking down of a melt into drops, said drops being caused to be ejected by the influence of centrifugal force and then solidify, including a
35 number of disk units (12) which are rotatable around an axis and a means of distributions to uniformly distribute the melt circumferentially on the disks c h a r a c t e r i s e d in that the disk units are composed of a number of disks arranged axially one on top of the other, whose cross sections include a radial inner section

with a mainly U-shaped recess in which a raised, essentially, L-shaped section of the closest underlying disk made in part with sufficient play therebetween to enable the radial ejection of a jet of liquid.

7. Apparatus according to claim 6 c h a r a c t e r i s e d in that the L-shaped
5 section of each disk is shaped such that the oblique, upright leg of the L-shape includes an oblique downwards -inwards directed surface (22) adapted to the jet of liquid and an integrated horizontal transition surface (23) connecting thereto.

8. Apparatus according to claim 6 or 7 c h a r a c t e r i s e d in that the
10 essentially U-shaped recess includes a radial, outer obliquely downward-outwards directed second surface (25) adapted to the jet of liquid.

9. Apparatus according to any of claims 6-8 c h a r a c t e r i s e d in that the angle of inclination of the first surface (22) is $45-55^\circ$ relative to the horizontal plane.

10. Apparatus according to any of claims 6-9 c h a r a c t e r i s e d in that the
15 angle of inclination of the second surface (25) is $65-75^\circ$ relative to the horizontal plane.

11. Apparatus according to any of claims 6-10 c h a r a c t e r i s e d in that a third surface (26) is arranged and integrated with the second surface (25) whereby the angle of inclination of said third contact surface is $<10^\circ$ relative to the horizontal
plane.

20 12. Powder consisting of an agglomerates of cemented carbide, cermets, ceramics or similar materials with abrasive wear resistance c h a r a c t e r i s e d in that they are made according to any of claims 1-11.

13. Powder according to claim 12 c h a r a c t e r i s e d in that it is in the form
25 of an agglomerate with a distribution of sizes with the ratio $d_{97}/d_{03} < 4$ where
 d_{97} = the grain size below which 97 % of the agglomerates is found and
 d_{03} = the grain size below which 3 % of the agglomerates is found.

14. Powder consisting of agglomerates of cemented carbide, cermets, ceramics
or similar materials with abrasive wear resistance c h a r a c t e r i s e d in that the
agglomerate has a distribution of size prior to any subsequent sieving with the ratio of
30 $d_{97}/d_{03} < 4$ where

d_{97} = the grain size below which 97 % of the agglomerates is found and
 d_{03} = the grain size below which 3 % of the agglomerates is found.

1 / 4

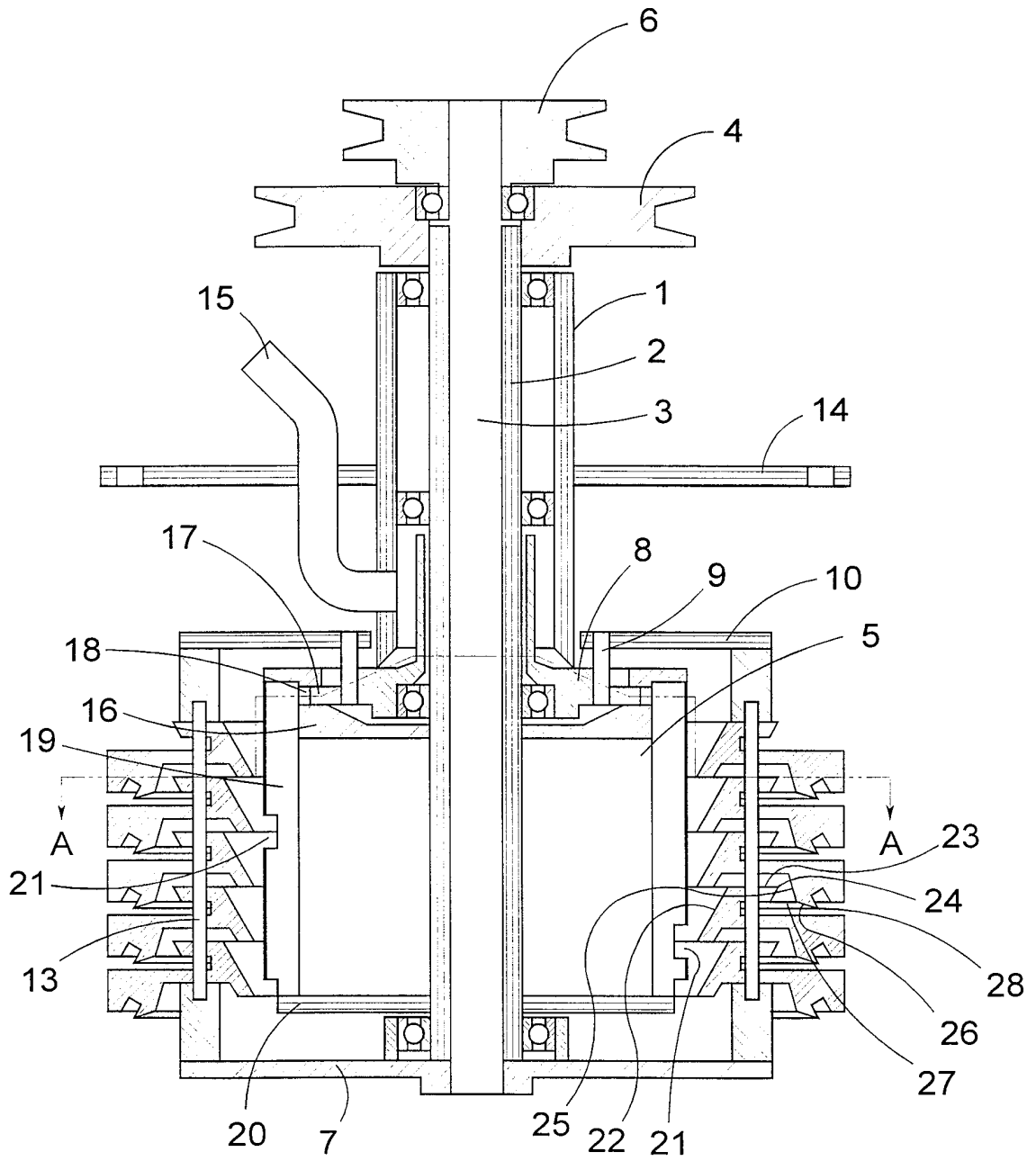


Fig. 1

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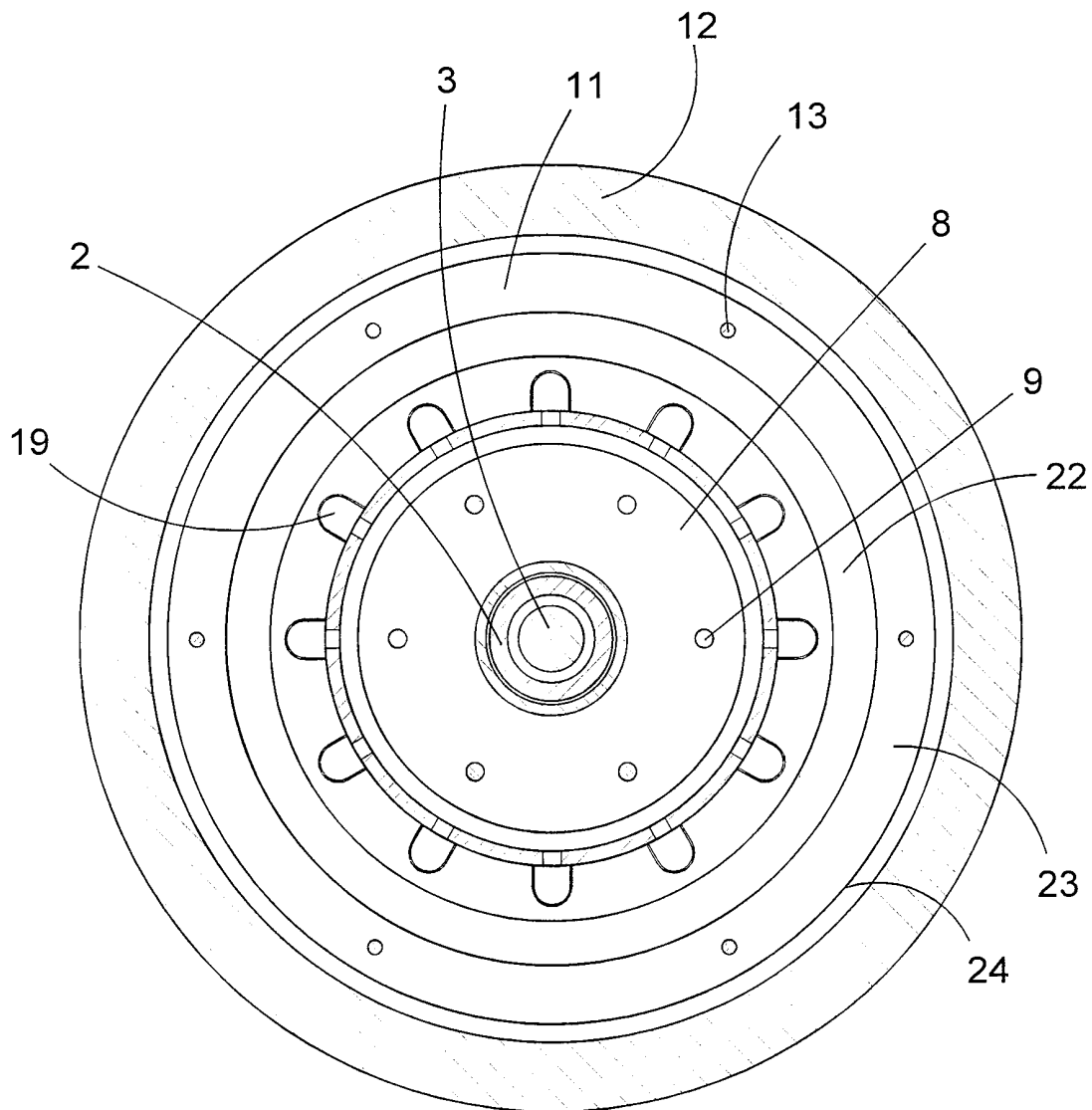


Fig. 2

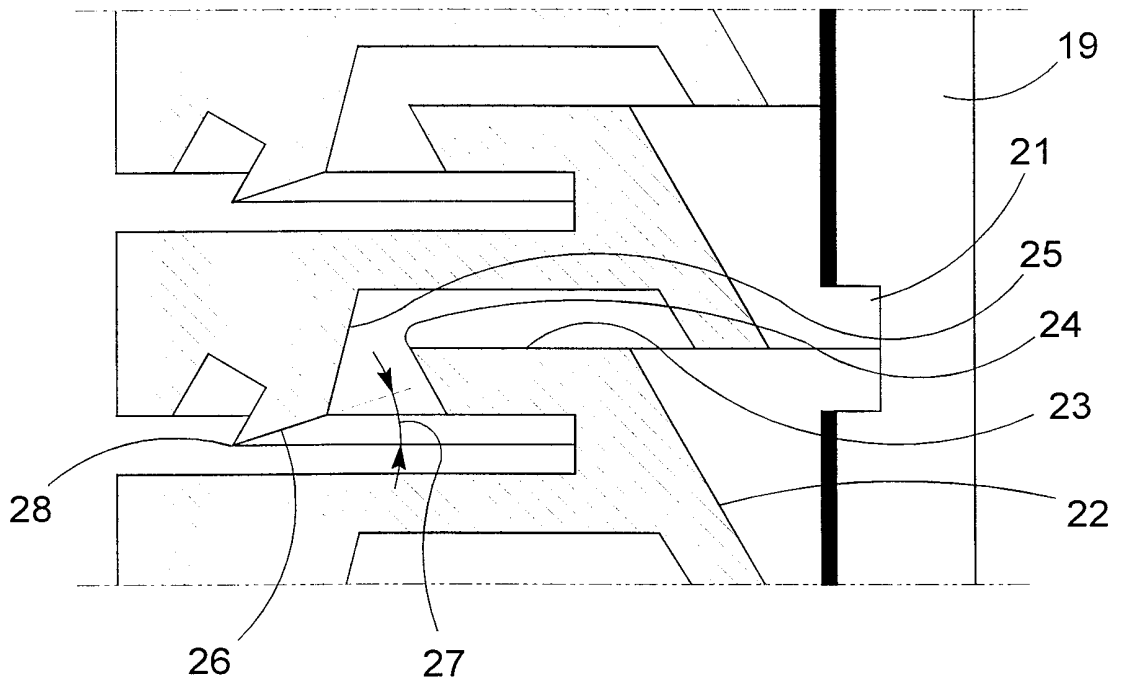


Fig. 3

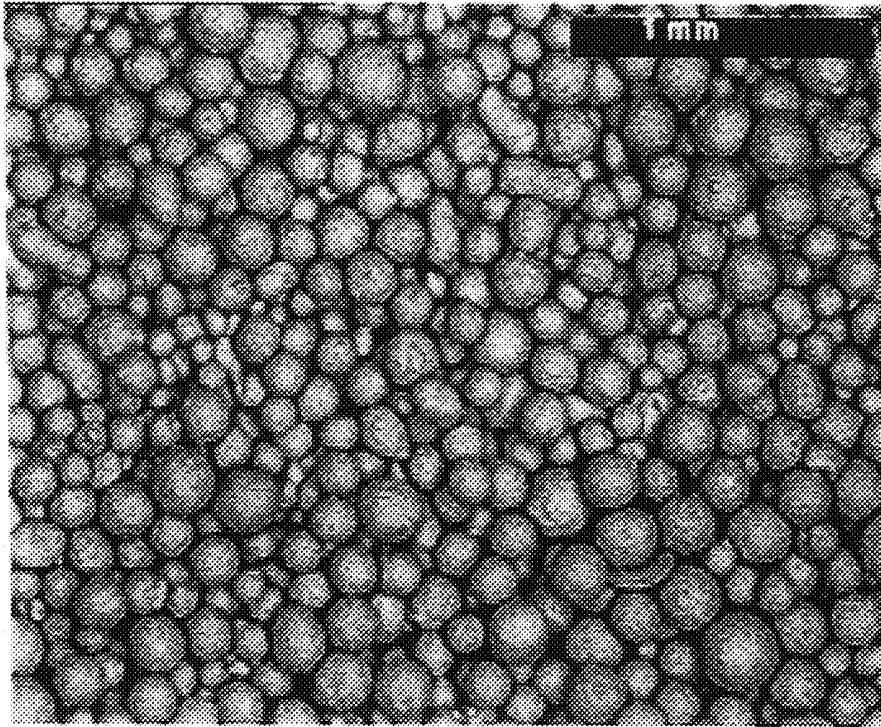


Fig. 4

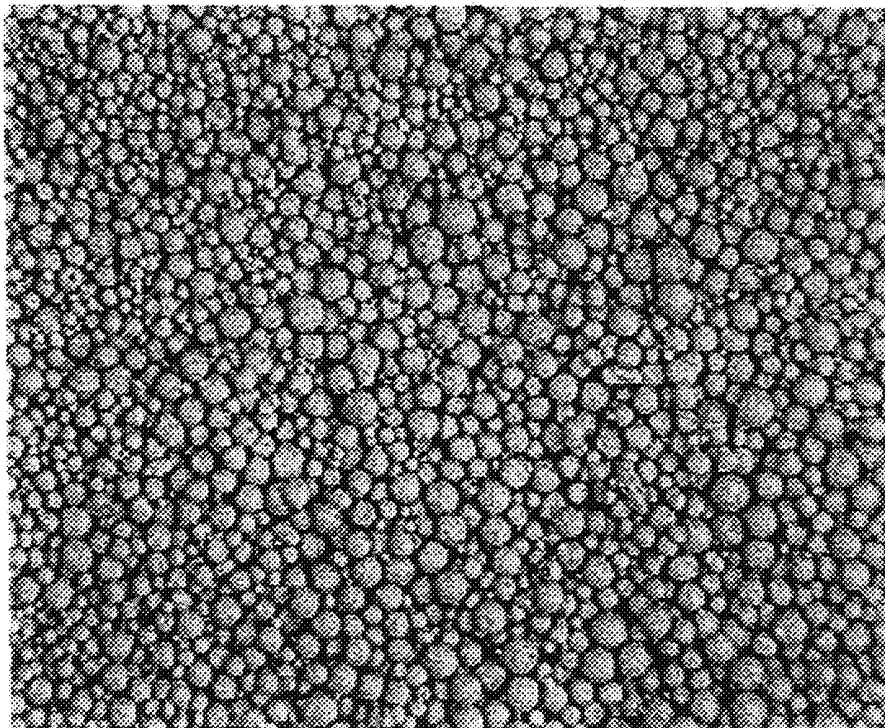


Fig. 5

INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 99/01698

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B22F 1/00, B22F 9/10, B01J 2/14, B05B 3/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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IPC7: B22F, B01J, B05B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4978069 A (RALF ANDERSSON ET AL), 18 December 1990 (18.12.90) --	1-14
A	DE 4308842 A1 (WALZEL, PETER), 22 Sept 1994 (22.09.94) -- -----	1-14

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INTERNATIONAL SEARCH REPORT
Information on patent family members

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International application No.
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