A ~~machine for moulding composite matter and a~~ method of producing ceramics-based composite

The invention relates to ~~a machine for moulding composite blocks and to~~ a method of producing composite based on ceramics, especially HAp, for implants.

Ceramic granules from hydroxyapatite HAp or from tricalcium phosphate TCP are used as materials for implants but due to their fragility and poor plasticity their application in surgery, especially oral and facial, is limited.

From patent description DE2238265 there is known a press having independently adjustable stages for manufacturing a multi-layered laminated product from powdered materials. The number of these stages depends on the number of powdered materials used. Each stage includes a piston whose stroke is guided by and limited by a cylindrical portion of an adjacent piston placed below the first one. The punch is hydraulically driven and its position is adjustable. The press is equipped with a feeder.

There are known methods of making the above mentioned ceramic materials more malleable by using water suspension of curdlan heated to 40°C.

From Polish patent PL 212866 there is known a method of producing bio-active composite based on calcium-phosphate bio-ceramics in the form of granules and which is mixed with curdlan in proportions according to a specific formula and then the mixture is heated in temperatures of 80-100°C.

The object of the invention ~~is a machine for moulding and ramming malleable matter and~~ a method of producing ceramics-based composite characterised by high mechanical endurance.

Unexpectedly, it turned out that reducing presence of air within composite structures to a minimum significantly improves their endurance.

~~According to the invention the forming machine is characterised in that it has a portioning assembly which comprises a dosing unit, favourably equipped with at least one linearly driven push rod, and a preliminary moulding unit, favourably equipped with at least one trough, whereby both these units are slidingly mounted on a plate of a carriage which is connected with linear drive. Above a mould form unit there is a guiding unit placed opposite a dosing unit. Above the guiding unit there is a pressing assembly slidingly mounted on a vertical frame. The pressing assembly is equipped with at least one piston rod having at its end a rammer. The piston rod is placed within a casing into which pneumatic pipes are connected. The portioning assembly is slidingly mounted parallely relative to the guiding unit. Drives of all the operating assemblies are connected with a control unit. The numbers of piston rods and of troughs are related to the number of the rammers and moulds.~~

~~Favourably, the rammer, which is cylindrically shaped, in its middle part has a narrowing, and on its side rings there are shallow furrows situated along the axis of the rammer.~~

Favourably, the mould form unit is mounted by means of clams and thus can be easily dismantled.

The machine guarantees that the matter for the composite is properly compacted. The efficient removal of the air from the matter is assured thanks to the design of the rammer, which allows air bubbles to move between the rammer and the mould. Multiple pressing devices and multiple mould forms accelerate the process of producing semi-finished products. Automated control ensures reproducible manufacture.

An example of the object of the invention is presented on diagrams, whereby Fig. 1 presents axonometric drawing of the moulding machine viewed from the carriage side, Fig. 2 – side view of the plate of the carriage together with the dosing assemblies, Fig. 3 – longitudinal sectional view along axis of piston rods, Fig. 4 – axonometric drawing of the machine viewed from the pressing assembly side, Fig. 5 – the rammer with partial section along line A, and Fig. 6 – the rammer front view.

A moulding machine having a bed **1** upon which a carriage **2** with a linear drive **3** on guides **4** is slidingly mounted. A dosing unit **6** with eight push rods **7** and a preliminary moulding unit **8**, which has eight troughs **9** running along axis of the push rods **7,** are both screwed onto a plate **5** of the carriage **2**. The preliminary moulding unit **8** is exchangeable and is mounted on the plate **5** of the carriage **2** using centering pins. The carriage **2** is connected with a primary linear drive **10**. On the bed **1** there is a guiding unit **11** installed having eight through-holes **12** situated vertically, and eight cylindrical horizontal holes **13** which are situated along the guiding unit **11** starting from its front wall, which is located at the side of the preliminary moulding unit, up to the through-holes **12**. The axis of the horizontal holes **13** flush with the axis of the push rods **7**. Under the guiding unit **11** there is a mould form unit **14** slidingly mounted and attached onto a frame **19** with clamps which make it easy to dismantle. The mould form unit **14** has eight cylindrical mould forms **15** whose axis flush with the axis of the through-holes **12**. Under the mould form unit **14** there is a slat **16** which has eight plugs **17** and which is supported by a pneumatic actuator **18**.

Mounted on the bed **1** there is the frame **19** onto which the pressing unit **20**, coupled with the secondary linear drive **21**, is fitted. The pressing unit **20** is equipped with eight pressing mechanisms **22**. The pressing mechanism **22** consists of a casing **24** into which two pneumatic pipes **23** are connected. In the casing **24** there is a piston rod **25** connected via a joint **26** and via a screw joint with a rammer **27**. The rammer **27** has a cylindrical shape with a narrowing **28** which creates two rings **29** on the sides. On the cylindrical surface of the rings **29** of the rammer **27** there are shallow furrows **30** having a minimal clearance relative to a diameter of a mould and situated along the axis of the rammer **27**. All the drives of the operating assemblies are connected with a control unit **31**.

All the elements of the assemblies that might get soiled, especially the preliminary moulding unit **8**, the push rods **7**, the mould form unit **14**, and the guiding unit **11,** can be easily dismantled for cleaning. The mould form unit **14** is made of chemically resistant material.

The machine functions as follows: The preliminary moulding unit **8,** with the troughs **9** thoroughly filled, is placed on the plate **5** of the carriage **2** and positioned with precision. On the front side, after lifting the pressing unit **20**, the mould form unit **14** is placed. The pneumatic actuator **18** supports the slat **16** with the plugs **17** which seal individual forming chambers **15**. After the pressing unit **20** is lowered, a pre-programmed process of ramming is

~~initiated running in cycles consisting of a feeding movement of the push rods **7**, which supply portions of composite to the through-holes **12** of the guiding unit **11**, and a synchronized with it movement of the rammers **27**. The rammers **27** and the push rods **7** move alternately and perpendicularly in relation to each other within the guiding unit **11**. When a portion of a composite is placed in the through-holes **12** the ramming is being executed by the rammer **27** moving three times. The cycle of ramming each individual portion of a composite is repeated 10-13 times. After the composite matter has been rammed, the pressing unit **20** is moved to the back and the mould form unit **14** is dismantled and then inlets of the mould forms **15** are plugged and next moved for a heat treatment.~~

~~The invention also relates to a method of producing ceramics-based calcium phosphate composite.~~

According to the invention a method for producing ceramics-based calcium phosphate composite is characterised in that into a water suspension, which contains 76-175 grams of β-1.3-glucan (hereinafter referred to as curdlan) per 1 litre of water, calcium phosphate ceramics in the form of porous granules with a total weight of 300-800 g, favourably from 415 to 800 g, is added and mixed until an optimum homogeneity is achieved and then preliminary moulding takes place whereby a preform trough is thoroughly filled with the matter and then the matter is pushed into a mould where each portion is rammed separately until consistency containing less than 15% air is achieved and, after accurate ramming, it undergoes 5-120 minutes of heat treatment in temperatures between 80-100°C, and after that a finished product is removed from the mould.

Favourably, the granules have sizes from 0.1 to 0.9 mm and an open porosity from 50% to 70%. The most favourable porosity is between 60 and 70%.

Favourably, prior the heat treatment, the mould containing the rammed matter is sealed with a plug or, otherwise, the air exposure of the rammed matter is restricted by using other means.

Examples

Example I

170 g of β-1.3-glucan was mixed with 1.000 ml of water and then 350 g of calcium phosphate ceramics HAp granules with a diameter of 0,3-0,4 mm were added and the ingredients were mixed until homogeneous consistency has been achieved. From the resulting matter a sample having about 6.5 cm^3 in volume was taken and the preform trough with a capacity of 5550 mm^3 was thoroughly filled with it. The remaining excess of the matter was removed. In the next step portions of the matter having a volume of about 0,4 cm^3 were being fed successively from the trough into a mould having a diameter of 13 mm and each portion was rammed three times with a force of 15 N. The obtained semi-finished product having a volume of 4 cm^3 was heat-treated for 25 minutes at 98°C and after that cooled to room temperature and then removed from the mould. It has been concluded that the homogeneous composite thus obtained had low plasticity and during a compression test the sample sustained deformation of 18% prior its destruction.

The composite has been dried for 12 hours at 40°C. The overall volume of pores (air content) in the dried sample of the composite, as measured with a method of computed micro

tomography (with threshold value for pores detection of 15 μm), amounted to 6.74% ± 1.15%. More than 90% of all pores in the sample were of the closed type.

Example II

For testing purpose 40 g of β -1.3-glucan mixed with 500 ml of water was used. 225 g of calcium-phosphate ceramics TCP granules with diameters of between 0,4-0,6 mm was added to the water suspension and then the components have been mixed until homogeneous matter was obtained.

From the obtained matter a sample having about 10 cm^3 in volume was taken and put into the preform trough with a capacity of 8.350 mm^3 which was thoroughly filled and the remaining excess of the matter was removed. In the next step portions of the matter having a volume of about 750 mm^3 each were being pushed out the trough into a mould with a diameter of 15 mm where each portion has been rammed three times with a force of 20 N. The obtained product having a volume of 7 cm^3 was heat-treated for 20 minutes at 93°C. The finished product, after having been cooled and after removing it from the mould, was examined under a microscope and no empty spaces resulting from the air remaining in the semi-finished product undergoing the moulding process have been discovered during this examination.

The overall volume of pores (air content) in the dried sample of the composite, as measured with a method of computed micro tomography (with threshold value for pores detection of 15 μm), amounted to 7.24% ± 1.11%. About 30% of those pores have diameters between 45-75 μm. No pores with a diameter more than 0.3 mm had been detected, which indicates highly homogeneous structure of the product and high efficiency of the moulding process performed by the machine hereby described.

Patent Claims

1. A machine for moulding, especially for manufacturing moulded semi-finished products for producing composite for implants, having a pressing unit and a mould form unit, characterised in that it has a portioning assembly comprising a dosing unit **(6)** and a preliminary moulding unit **(8)**, whereby above the mould form unit **(14)** there is a guiding unit **(11)** situated opposite the dosing unit **(6)** and above the aforementioned guiding unit **(11)** there is a pressing assembly **(20)** slidingly mounted on a vertical frame **(19)** and equipped with at least one piston rod **(25)** with a rammer **(27)** at its end, whereby the piston rod **(25)** is placed in a casing **(24)** into which pneumatic pipes **(23)** are connected and, moreover, the portioning assembly is mounted slidingly in parallel plane relative to the guiding unit **(11)**, and whereby all the drives of operating units are connected with a control unit **(31)**.
2. The machine for moulding in accordance with claim 1, characterised in that the rammer **(27)** has a cylindrical shape with a narrowing **(28)** and on its side rings **(29)** there are shallow furrows **(30)** running along the axis of the rammer **(27)**.
3. The machine for moulding in accordance with claim 1, characterised in that the portioning assembly is mounted on a plate of a carriage **(2)** equipped with a linear drive **(3)**.
4. The machine for moulding in accordance with claim 1, characterised in that the preliminary moulding unit **(8)** has at least one trough **(9)**.
5. The machine for moulding in accordance with claim 1, characterised in that the mould form unit **(14)** is fastened with clams and thus detachable.
6. The machine for moulding in accordance with claim 1, characterised in that the dosing unit **(6)** is equipped with at least one push rod **(7)** moved by a linear drive **(3)**.
7. The machine for moulding in accordance with claim 1, characterised in that the number of push rods **(7)** and troughs **(8)** relates to the number of piston rods **(25)** and mould forms **(15)**.

8. 1.A method of producing composite based on calcium phosphate ceramics mixed with curdlan, characterised in that into a water suspension containing 76-175 grams of β-1,3-glucan (hereinafter referred to as curdlan) per one litre of water calcium phosphate ceramics in the form of porous granules weighing 300-800 g in total, favourably from 415 to 800 g, are added and mixed until an optimum homogeneity is achieved and then preliminary moulding takes place whereby a preform trough is thoroughly filled with the matter and after that the matter is pushed into a mould where each portion of the matter is rammed separately until consistency containing less than 15% air is achieved and, after accurate ramming, the matter together with the mould is being kept for 5-120 minutes in temperatures between 80-100°C, and after that the finished product is removed from the mould.

9. 2.The method in accordance with claim 8 1, characterised in that the granules have a diameter of between 0.1 to 0.9 mm and an open porosity ratio of between 50% and 70%.

10. 3.The method in accordance with claim 8 2, characterised in that, favourably, the porosity of the granules is between 60% and 70%.

~~11.~~ 4.The method in accordance with claim ~~8~~ 1, characterised in that the mould containing the rammed matter, prior to heat treatment, is plugged or, alternatively, the air exposure of the rammed matter is restricted by using other means.

Abstract

The inventions solve the problem of ~~designing a machine for producing moulded composite parts that are homogeneously structured and the problem of~~ finding a method for producing composite for implants.

~~The essence of the design of the machine consists in that it has a portioning assembly, whereby above a mould form unit **(14)** there is a guiding unit **(11)** located opposite a dosing unit **(6)** and above the aforementioned guiding unit **(11)** there is a pressing assembly **(20)** slidingly mounted on a vertical frame **(19)** and which is equipped with at least one piston rod **(25)** with a rammer **(27)** at its end, whereby the piston rod **(25)** is placed within a casing **(24)** into which pneumatic pipes **(23)** are connected.~~

The essence of the method consists in that a preliminary moulding is being performed by thoroughly filling a preform trough and then the matter is being pushed out of the preform trough in portions and then each portion is rammed separately until consistency containing less than 15% air is achieved.

[Fig.1]