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(71) Applicant: Matteazzi, Paolo 31100 Treviso (IT)

(72) Inventors:

- MATTEAZZI, Paolo 31100 Treviso (IT)
- SALVADOR, Marco 31044 Montebelluna (TV) (IT)
- (74) Representative: Lunardelli, Alessandro et al Dragotti & Associati S.r.l.
   Via Paris Bordone, 9 31100 Treviso (IT)

# (54) MECHANO-CHEMICAL REACTOR FOR PHYSICAL OR PHYSICAL-CHEMICAL TREATMENT OF SOLID AND/OR LIQUID SUBSTANCES

(57) Mechano-chemical reactor (1) for physical or physical-chemical treatment of substances in the solid and/or liquid state, comprising a load-bearing structure (2) and at least one oscillating mass or jar (6A) with a reciprocating movement supported by the load-bearing structure (2) and defining at least one confined environment (14A) inside which the substances to be treated and a plurality of grinding elements are arranged. The at least one oscillating mass or jar (6A) is subjected to a reciprocating movement along the direction of a vertical

axis (V1) so as to carry out the treatment on the substances by means of the grinding elements. The reactor (1) also comprises means (5) for driving the at least one oscillating mass or jar (6A) and an elastic system (4) for compensating for a part of the inertial forces which are generated by the oscillations of the at least one oscillating mass or jar (6A). The elastic system (4) comprises elastic means (18) mounted on the load-bearing structure (2) and extending along respective axes (V2) parallel to the vertical axis (V1).

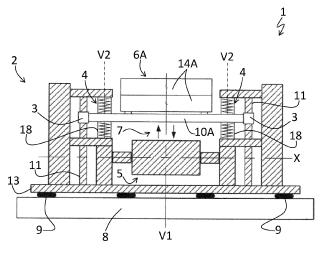


Fig. 1

# Description

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**[0001]** The present invention relates to a mechano-chemical reactor in which the kinetic energy of a plurality of grinding elements is used to subject one of more substances in the solid and/or liquid state to a treatment designed to modify the physical or chemical characteristics thereof.

**[0002]** In particular, the treatment is performed inside at least one confined environment, such as a closed chamber, inside which the substances in the solid and/or liquid state to be treated are loaded beforehand.

**[0003]** From the prior art, it is known to use so-called high-energy reactors or mills which use the kinetic energy, which is usually higher than 400 W/dm³, of grinding bodies or elements to subject the substances to physical or chemical-physical treatments.

**[0004]** The high-energy mills are used mainly, although not exclusively, for the production of nanomaterials, namely materials formed by particles or grains which have dimensions typically smaller than 100 nanometres, i.e. less than 10<sup>-7</sup> metres.

**[0005]** The article written by J. Sidor, "Mechanical devices for production of metallic, ceramic-metallic alloys or nanomaterials, in Archives of Metallurgy and Materials of the Polish Academy of Sciences, n. 3/2007" is a presentation of numerous devices of the aforementioned type which are already in use.

**[0006]** However, as can be deduced from some data tables contained in this article, many of the known high-energy reactors or mills have a low production capacity and have a limited flexibility, since they are not suitable for uses other than by research laboratories and by limited industrial production facilities (pilot production plants).

**[0007]** On the other hand, an example of a high-energy mill designed to have a high production capacity is described in European Patent EP0665770.

**[0008]** EP0665770 describes a high-energy mill comprising at least one oscillating mass or jar intended to be filled with balls or other grinding elements and with given quantities (batches) of the substances to be treated.

**[0009]** The jar is subjected to a reciprocating movement, i.e. oscillations, along an axis corresponding to the geometric axis of the jar, namely along a vertical axis.

**[0010]** Moreover, during the operation of the mill, an elastic system compensates at least for a part of the inertial forces which are generated during the oscillations of the jar and which have a sinusoidal progression.

**[0011]** The elastic system preferably comprises two cylindrical springs which are arranged in contact with the top base and the bottom base of the jar, respectively.

[0012] Another example of a reactor or mill of the aforementioned type is described in the patent IT1403457, in which the elastic system comprises one or more flexible unidirectional elements (straight bars) or bidirectional elements (flat polygon) which extend substantially perpendicularly with respect to the direction of the oscillations.

**[0013]** Said reactor also comprises hinging means for coupling the flexible elements to the oscillating mass and to the load-bearing structure, these hinging means preventing the deformation of the flexible elements, in particular the bars, following the oscillations of the oscillating mass.

**[0014]** In order to increase the production capacity, the mill may also comprise a plurality of jars or oscillating masses which are in phase-opposition with each other.

[0015] Despite the fact that the aforementioned solutions are widely used and well-established in the sector, they are not without a number of drawbacks.

**[0016]** In particular, the reactors or mills of the type described above have a limited productivity and a reduced stability of the various components. Furthermore, the reactors or mills described above have particularly high maintenance costs.

**[0017]** A further drawback, in particular in connection with the technical solution described in IT1403457, is associated with the fact that the vertical axis along which the reciprocating movement of the jar takes place is subject to transverse oscillations due to the contractions of the flexible elements of the elastic system.

[0018] This latter drawback generates a movement induced in the grinding elements which reduces the operating efficiency and increases the energy consumption of the system.

**[0019]** The main object of the present invention is to provide a mechanochemical reactor which is further improved with respect to the prior art and which is able to solve the aforementioned drawbacks.

**[0020]** A particular task of the present invention is to provide a mechano-chemical reactor which has a productivity substantially improved compared to the reactors or mills known from the prior art. A further task of the present invention is to provide a mechano-chemical rector which is able to reduce the transverse oscillations acting on the transmission means and therefore on the vertical axis of the oscillating mass.

**[0021]** A further task of the present invention is to provide a mechano-chemical reactor which has lower maintenance costs compared to the reactors known from the prior art.

**[0022]** A further task of the present invention is to provide a mechano-chemical reactor which is able to improve the stability of the components during operation.

**[0023]** Another task of the present invention is to provide a mechano-chemical reactor which is able to improve the kinetics of the process.

**[0024]** A further task of the present invention is to provide a mechano-chemical reactor able to operate in a continuous manner, namely which is able to treat not only single batches, but also indefinite quantities of substances in the solid and/or liquid state, with the possibility of reaching particularly high production capacities.

[0025] A further task of the present invention is to provide a mechano-chemical reactor which allows the moving masses to be increased.

**[0026]** The main object and tasks described above are achieved with a mechano-chemical reactor for the physical or physical-chemical treatment of solid and/or liquid substances in accordance with Claim 1.

**[0027]** In order to illustrate more clearly the innovative principles of the present invention and its advantages compared to the prior art, an example of embodiment of the mechano-chemical reactor according to the present invention will be described below with the aid of the attached figures. In particular, in the figures:

- Figure 1 is a schematic cross-sectional view of the mechano-chemical reactor in accordance with a first embodiment;
- Figure 2 is a schematic cross-sectional view of the mechano-chemical reactor in accordance with a second embodiment.

**[0028]** The present description, provided only by way of a non-limiting example of the scope of protection of the invention, relates to a mechano-chemical reactor denoted overall by the reference number 1.

**[0029]** Advantageously, the reactor 1 according to the present invention is intended for the physical or chemical-physical treatment of substances in the solid and/or liquid state, as will become clear from the continuation of the present description.

[0030] In particular, the further improved mechano-chemical reactor 1 according to the present invention allows the production of nanomaterials with physical and chemical-physical characteristics which are transformed with respect to the starting substances; these characteristics comprise for example the chemical combination state of the elements, the molecular weights (when the materials are polymers), the aggregation state and the size of the crystals (when the materials are inorganic crystalline materials), the alloying and solid solution state and the mixing states of different phases.

[0031] In this connection, the mechano-chemical reactor 1 according to the present invention uses the kinetic energy of a plurality of grinding elements or bodies subjected to a reciprocating oscillating movement, as described in detail below. The grinding elements are not visible in the attached figures.

**[0032]** The mechano-chemical reactor 1 according to the present invention may also be used in technical fields different from the field for the production of nanomaterials, such as the field for mixing substances, the crushing field, the chemical synthesis and metallurgical field and the field for the production of high-viscosity dispersed liquids.

[0033] In the most general embodiment, the mechano-chemical reactor 1 comprises:

a load-bearing structure 2;

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- at least one oscillating mass or jar 6A supported by said load-bearing structure 2 and defining at least one confined environment 14A inside which the substances to be treated and a plurality of grinding elements are arranged, the at least one oscillating mass or jar 6A being subjected to a reciprocating movement along the direction of a vertical axis V1 so as to carry out the treatment on the substances by means of the grinding elements;
- means 5 for driving the at least one oscillating mass or jar 6A.

**[0034]** The main difference between the embodiments shown in Figure 1 and Figure 2, respectively, relates to the number of oscillating masses provided in the reactor.

**[0035]** In particular, the first embodiment of Figure 1 has a single oscillating mass or jar 6A weighing more than 500 kg and preferably close to 1100 kg; the second embodiment according to Figure 2 has two oscillating masses or jars 6A, 6B, weighing overall more than 2000 kg and preferably close to 4000 kg.

**[0036]** In the second embodiment, both the oscillating masses 6A, 6B are connected to the driving means 5 which are in phase-opposition and have a mutual counter-phase movement. The oscillating masses or jars 6A, 6B of the second embodiment define respective confined environments 14A, 14B.

[0037] Advantageously, the jar 6A or the jars 6A, 6B have a substantially symmetrical reciprocating movement.

**[0038]** Preferably, the load-bearing structure 2 comprises a first rigid base 13 and a second rigid base 8 which rests on the ground. A plurality of damping elements 9 are arranged between the two rigid bases 13, 8.

**[0039]** The damping elements 9 are able to absorb the vibrations acting on the reactor 1 overall and a small amount of the inertial forces acting along the load-bearing structure 2.

**[0040]** Furthermore, the load-bearing structure 2 comprises a plurality of uprights or vertical posts 11, these uprights 11 being secured to the first rigid base 13 and extending along respective vertical directions perpendicular to the ground. **[0041]** Conveniently, the at least one oscillating mass or jar 6A or the oscillating masses or jars 6A, 6B are fixed onto respective supports 10A, 10B.

[0042] The reciprocating oscillations of the at least one oscillating mass 6A or of the oscillating masses 6A, 6B along

the vertical axis V1 have a frequency greater than 10 Hz, preferably close to 15 Hz, and an amplitude greater than 20 mm, preferably close to 30 mm.

**[0043]** The inertial forces generated by the oscillations therefore have a sinusoidal progression. Advantageously, the at least one jar 6A or the jars 6A, 6B are made of a wear-resistant steel, for example Hardox<sup>®</sup>, and have the form of a flattened cylinder with its geometric axis which coincides with the vertical oscillation axis V1.

**[0044]** However, other solutions of materials (for example Ni, Ti, Cu or other alloys) which are chemically compatible with the products being processed and/or for lightening the jar 6A or the jars 6A, 6B may be advantageously used.

**[0045]** Moreover, the shape of the jars 6A, 6B may also be different from the shape indicated above, without thereby departing from the scope of protection of the present invention.

**[0046]** Conveniently, the walls of the at least one jar 6A or the jars 6A, 6B comprise at least one pair of openings, not shown in the attached figures, for supplying the solid and/or liquid substances to be treated and for discharging the products following the chemical or chemical-physical treatment. These openings may be provided with valves.

**[0047]** The jars 6A, 6B may also incorporate auxiliary devices of the conventional type, not shown in the attached figures, such as heat exchangers, vacuum pumps, etc., as well as control devices, for example temperature and pressure sensors and also vibration sensors.

[0048] Owing to these auxiliary devices, it is ensured that the treatment of the substances loaded into the jars 6A, 6B takes place in predetermined and predefined environmental conditions.

**[0049]** The grinding elements loaded into the at least one confined environment 14A comprise preferably a plurality of spheres or spheroids (balls) which are made of a material which is particularly resistant to corrosion and wear, such as chrome steel, namely made using materials which are chemically compatible with the products (similar to the material of the jar 6A or the jars 6A, 6B).

**[0050]** Furthermore, each oscillating mass or jar 6A, 6B may define a plurality of confined environments 14A, 14B which are in fluid communication with each other for the passage of the substances to be treated.

**[0051]** These oscillating masses or jars 6A, 6B are known as multiple jars and have characteristics similar to those indicated above with reference to the known reactors.

**[0052]** The confined environments 14A, 14B may be connected together for the supplying and discharging of the substances to be treated and the products obtained following the treatment in succession between one environment and another.

[0053] Moreover, each of the confined environments 14A, 14B comprises a predetermined number of grinding elements located inside it.

**[0054]** As shown in the attached figures, the confined environments 14A, 14B may have an axis of development perpendicular to the vertical axis V1 and may be arranged above one another. Irrespective as to the number of confined environments 14A, 14B formed in the oscillating masses 6A, 6B, the operation of the reactor 1 in this respect may be performed using three different operating modes, namely:

- discontinuously, with the reciprocating movement of the oscillating mass 6A or the oscillating masses 6A, 6B interrupted or suspended during the supplying of the substances to be treated or the discharging of the products;

- semi-continuously, namely with the supplying of the substances and the discharging of the products performed during the reciprocating movement of the oscillating masses 6A, but in different phases;

- continuously, namely with the supplying and discharging performed continuously during the reciprocating movement of the oscillating mass 6A or oscillating masses 6A, 6B.

**[0055]** It is understood that the treatments of the substances in the reactor 1 may be controlled manually or automatically based on the signals provided by sensors or other devices for monitoring the temperature and pressure values in the at least one closed chamber of the oscillating mass. These monitoring devices are not shown in the attached figures.

**[0056]** As shown in the aforementioned figures, the supports 10A, 10B introduced above occupy a greater area than the corresponding jars 6A, 6B.

**[0057]** With particular reference to the first embodiment shown in Figure 1, the support 10A of the jar 6A is located in a position closer to the ground than the jar 6A along the vertical axis V1.

**[0058]** With particular reference to the second embodiment shown in Figure 2, the further support 10B is located on a position further from the ground than the corresponding jar 6B along the vertical axis V1.

**[0059]** The driving means 5, shown schematically in the attached figures and also defined as movement means, may comprise a connecting rod/crank mechanism or a hydraulic system or a linear or magnetic motor.

**[0060]** Moreover, the driving means 5 extend along a substantially horizontal axis X perpendicular to the vertical axis of oscillation V1 of the oscillating mass 6A or the oscillating masses 6A, 6B.

[0061] In both the embodiments of the present invention, the driving means 5 are supported by the load-bearing structure 2, as can be seen in Figures 1 and 2.

[0062] Advantageously, the driving means 5 are connected to the oscillating mass 6A of the first embodiment or to

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both the oscillating masses 6A, 6B of the second embodiment, by means of a suitable member or suitable transmission means 7, indicated schematically in the figures by means of a pair of arrows.

**[0063]** As already mentioned, in the second embodiment, both the oscillating masses 6A, 6B are moved with a reciprocating movement in phase opposition by means of unitary driving units 5 connected independently to either oscillating mass by means of the transmission means or member 7.

[0064] The transmission means or member 7 may be of the type described in IT1403457.

**[0065]** The mechano-chemical reactor 1 according to the present invention also comprises an elastic system 4, which is preferably pre-compressed, for compensating for a part of the inertial energy generated by the oscillations of the at least one oscillating mass 6A or of both the oscillating masses 6A, 6B where present.

[0066] In particular, the elastic system 4 allows the at least one oscillating mass or jar 6A, or both the oscillating masses or jars 6A, 6B, to be connected in a non-rigid manner to the load-bearing structure 2 of the reactor 1.

**[0067]** In accordance with a particular aspect of the invention, the elastic system 4 comprises elastic means 18 mounted on the load-bearing structure 2; these elastic means 18 extend along respective axes V2 which are parallel to the vertical oscillation axis V1 and are preferably radially staggered with respect to the vertical axis V1.

**[0068]** With reference to this latter characteristic feature, it is understood that the axes V2 of the elastic means 18 are not coaxial with the vertical oscillation axis V1.

**[0069]** With the elastic system 4 thus configured it is possible to obtain dynamic compensation of the inertial forces, which have a sinusoidal progression and are generated by the oscillating masses 6A, 6B, greater than 70%.

**[0070]** Moreover, by means of the aforementioned configuration of the mechanochemical reactor 1 it is possible to obtain an improvement in the productivity equal to about 20% compared to the known reactors and a reduction in the maintenance costs of between 16% and 20% compared to the known reactors.

**[0071]** The elastic means 18 are mounted on the load-bearing structure 2 by means of coupling and fixing means of the known type, which will not be further described in the present description. Conveniently, and as illustrated more clearly in the attached figures, the elastic means 18 act on the support 10A or the supports 10A, 10B of the oscillating masses 6A. 6B.

**[0072]** Advantageously, the reactor 1 according to the present invention comprises a plurality of elastic means 18 which act on the ends of each support 10A, 10B and have respective axes V2 which are radially staggered with respect to the vertical axis V1.

**[0073]** Said plurality of elastic means 18 are formed preferably by helical springs, which are preferably pre-compressed, and are made of steel or other metal alloys, as shown more clearly in Figures 1 and 2.

**[0074]** Conveniently, the mechano-chemical reactor 1 according to the present invention may also comprise guiding means 3 associated with the at least one oscillating mass or jar 6A or both the oscillating masses or jars 6A, 6B according to the second embodiment.

**[0075]** The guiding means 3 are coupled with the load-bearing structure 2 so as to slidably support the at least one oscillating mass or jar 6A or the oscillating masses or jars 6A, 6B on the load-bearing structure 2. In the attached figures, the guiding means 3 are shown in schematic form.

[0076] The guiding means 3 are chosen from the group comprising bushes, bearings and carriages or are formed by sliding shoes.

**[0077]** Advantageously, the guiding means 3 are mounted on the support 10A or on the supports 10A, 10B, where provided, of the oscillating masses 6A, 6B and are slidably coupled onto the vertical posts or uprights 11 of the load-bearing structure 2.

**[0078]** Furthermore, the guiding means 3 are mounted on the load-bearing structure 2 so as to be slidable along a direction parallel to the vertical axis V1 of the at least one oscillating mass 6A or of the oscillating masses 6A, 6B.

**[0079]** With particular reference to the second embodiment of the reactor 1 according to Figure 2, each oscillating mass or jar 6A, 6B (with the respective support 10A, 10B) has, in addition to the respective elastic means 18, also respective guiding means 3 for slidable supporting on the load-bearing structure 2.

**[0080]** Furthermore, according to this embodiment, the guiding means 3 of the two oscillating masses 6A, 6B slide along the same vertical posts 11 in staggered positions along the vertical axis V1.

**[0081]** In order to highlight the productive capacity of the mechano-chemical reactor 1 according to the present invention, the table below shows a comparison of the characteristics and performance results of a first and second embodiment, respectively, of a reactor prototype according to the present invention with the reactor model forming the subject of the prior art document IT1403457:

Characteristics	First embodiment	Reactor model IT1403457		
Oscillating mass	1,100 kg	484 kg		
Motor power	20 kW	11 kW		

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(continued)

Characteristics	First embodiment	Reactor model IT1403457	
Oscillation frequency	15 Hz	15 Hz	
Oscillation amplitude	30 mm	30 mm	
Inertial forces	15,000 kg	6,400 kg	
Typical grinding batch	5 dm <sup>3</sup>	2 dm <sup>3</sup>	
Characteristics	Second embodiment	Reactor model IT1403457	
Oscillating mass	4,000 kg	4,000 kg	
Motor power	70 kW	60 kW	
Motor power Oscillation frequency	70 kW 15 Hz	60 kW 15 Hz	
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Oscillation frequency	15 Hz	15 Hz	

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**[0082]** From the above description it is now clear how the mechano-chemical reactor according to the present invention is able to achieve advantageously the predefined objects.

**[0083]** In particular, the use of elastic means which have respective axes parallel and radially staggered with respect to the vertical oscillation axis, in combination with the use of the guiding means, is able to reduce the oscillations acting on the transmission means along the vertical axis. Consequently, the mechano-chemical reactor according to the present invention has a greater productivity compared to the known reactors and at the same time is able to reduce the maintenance costs.

**[0084]** Moreover, the configuration of the reactor described above is able to improve the process kinetics and the stability of the mechanical components and increase the moving masses. Obviously, the above description of embodiments applying the innovative principles of the present invention is provided by way of example of these innovative principles and must therefore not be regarded as limiting the scope of the rights claimed herein.

#### Claims

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- 1. Mechano-chemical reactor (1) for physical or physical-chemical treatment of solid and/or liquid substances, comprising:
  - a load-bearing structure (2);
  - at least one oscillating mass or jar (6A) with a reciprocating movement supported by said load-bearing structure (2) and defining at least one confined environment (14A) inside which the substances to be treated and a plurality of grinding elements are arranged, said at least one oscillating mass or jar (6A) being subjected to a reciprocating movement along the direction of a vertical axis (V1) so as to perform the treatment on the substances by means of the grinding elements;
  - means (5) for driving said at least one oscillating mass or jar (6A);
  - an elastic system (4) for compensating for a part of the inertial forces which are generated by the oscillations of said at least one oscillating mass or jar (6A);

**characterized in that** said elastic system (4) comprises elastic means (18) mounted on said load-bearing structure (2) and extending along respective axes (V2) parallel to said vertical axis (V1).

- **2.** Reactor (1) according to Claim 1, **characterized in that** the at least one oscillating mass (6A) weighs more than 500 kg.
- 3. Reactor (1) according to Claim 1, **characterized in that** it comprises two oscillating masses or jars (6A, 6B) which are connected to said driving means (5) in phase opposition and defining respective confined environments (14A, 14B).

- 4. Reactor (1) according to the preceding claim, **characterized in that** said oscillating masses (6A, 6B) have an overall weight of more than 2000 kg.
- 5. Reactor (1) according to either one of Claims 3 or 4, **characterized in that** each of said oscillating masses or jars (6A, 6B) has respective elastic means (18).
  - **6.** Reactor (1) according to any one of the preceding claims, **characterized in that** the axes (V2) of said elastic means (18) are radially staggered with respect to said vertical axis (V1).
- 7. Reactor (1) according to any one of the preceding claims, **characterized in that** sad at least one oscillating mass or jar (6A) or said oscillating masses or jars (6A, 6B) are fixed onto respective supports (10A, 10B), said elastic means (18) acting on said supports (10A, 10B).
- 8. Reactor (1) according to any one of the preceding claims, **characterized in that** it comprises guiding means (3) associated with said at least one oscillating mass or jar (6A) or with said oscillating masses or jars (6A, 6B) and coupled to said load-bearing structure (2) for slidably supporting said at least one oscillating mass or jar (6A) or said oscillating masses or jars (6A, 6B) on said load-bearing structure (2).
- **9.** Reactor (1) according to the main claim, **characterized in that** said guiding means (3) are chosen from the group comprising bushes, bearings and carriages or consist of sliding shoes.
  - **10.** Reactor (1) according to any one of the preceding claims, **characterized in that** said elastic means (18) comprise helical springs, preferably made of steel or other metal alloys.
- 25 11. Reactor (1) according to any one of the preceding claims, characterized in that said driving means (5) comprising a connecting rod/crank mechanism or a hydraulic system or a linear or magnetic motor.

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- **12.** Reactor (1) according to any one of the preceding claims, **characterized in that** each oscillating mass (6A, 6B) define a plurality of confined environments (14A, 14B) in fluid communication with each other for the passage of the substances to be treated.
- **13.** Reactor (1) according to any one of the preceding claims, **characterized in that** the reciprocating movement of the at least one oscillating mass (6A) or of the oscillating masses (6A, 6B) has a frequency greater than 10 Hz, preferably close to 15 Hz, and an amplitude greater than 20 mm, preferably close to 30 mm.
- **14.** Reactor (1) according to any one of the preceding claims, **characterized in that** the dynamic compensation of the inertial forces is greater than 70%.
- **15.** Reactor (1) according to any one of the dependent claims, **characterized in that** said at least one jar (6A) or said jars (6A, 6B) are subjected to a substantially symmetrical reciprocating movement.

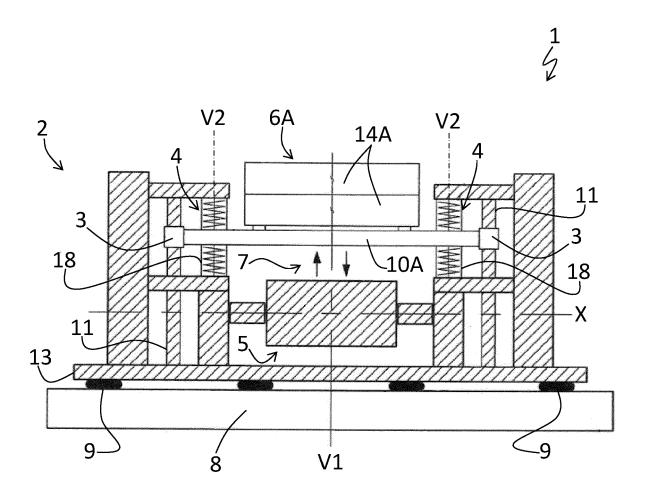


Fig. 1

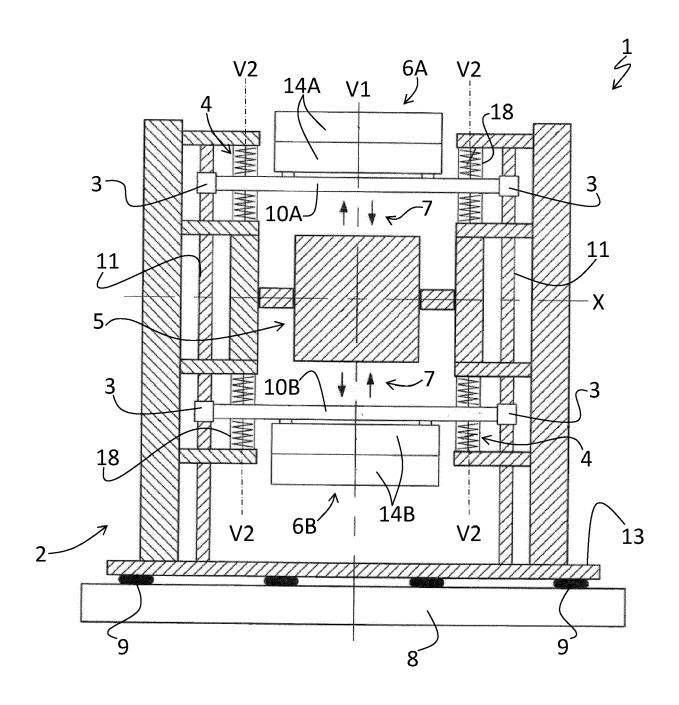


Fig. 2



# **EUROPEAN SEARCH REPORT**

**Application Number** 

EP 23 17 5047

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	DOCUMENTS CONSIDERED			
Category	Citation of document with indicatio of relevant passages	n, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
x	US 2 468 515 A (THOMAS 26 April 1949 (1949-04- * column 9, line 70 - c figures 6-8 * * column 12, lines 67-7 * column 6, line 68 - c	26) olumn 11, line 39; 1 *	1–15	INV. B02C17/14
x	DE 35 00 211 A1 (RETTEN 10 July 1986 (1986-07-1 * page 14, paragraph 3 paragraph 4; claims 1,2	0) - page 22,	1-15	
				TECHNICAL FIELDS SEARCHED (IPC)
	The present search report has been di	rawn up for all claims  Date of completion of the search		Examiner
	Munich	25 September 2023	Lau	ırim, Jana
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EP 23 17 5047

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10	cite	Patent document ed in search report	ı	Publication date		Patent family member(s)	Publication date
	us	2468515	A	26-04-1949	NONE		
15	DE 	3500211 	A1	10-07-1986	NONE		
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#### REFERENCES CITED IN THE DESCRIPTION

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