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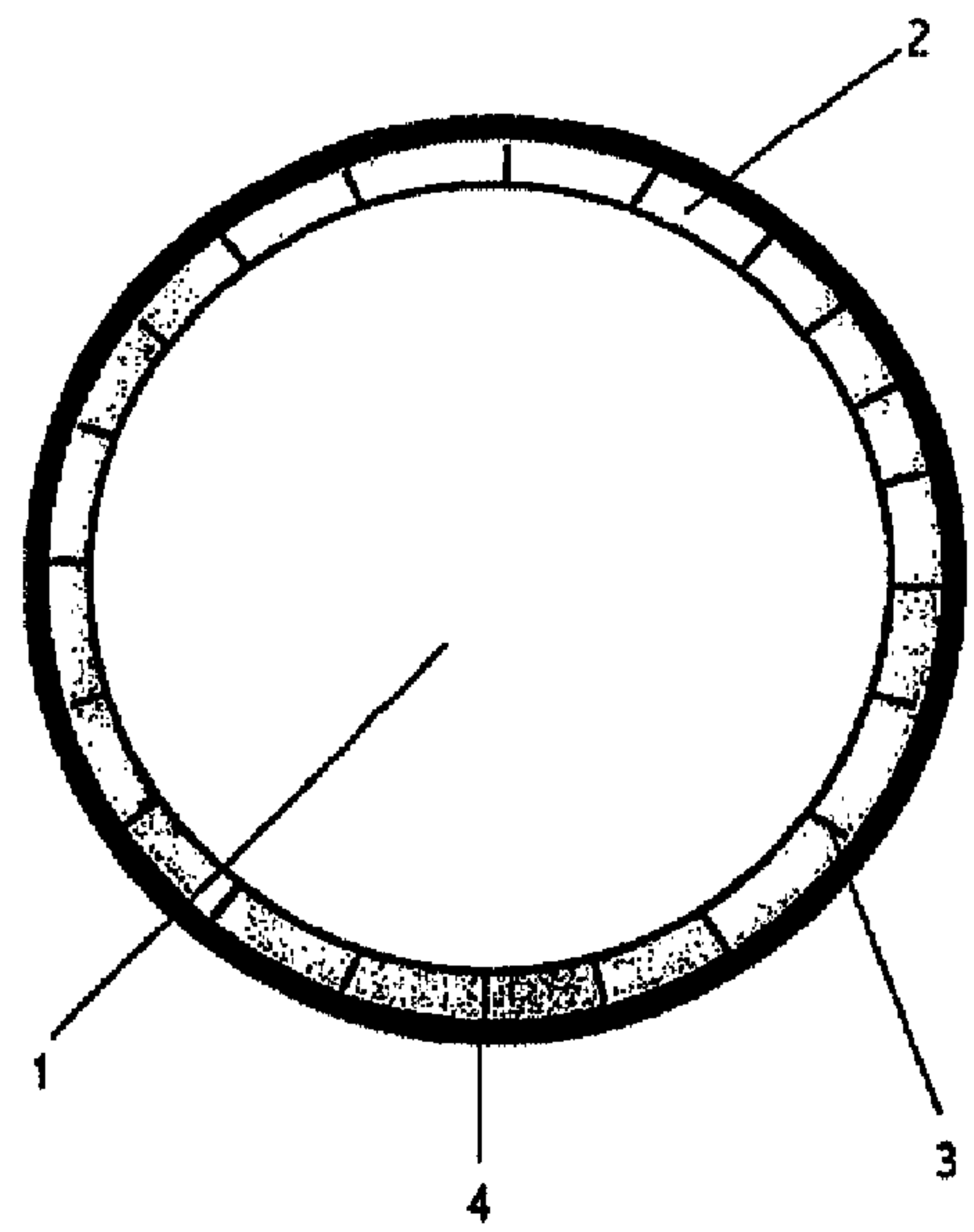
(54) Title: METHOD FOR ENCAPSULATING SOLDER METAL POWDERS AND SOLDER METAL POWDERS PRODUCED ACCORDING TO THIS METHOD



(57) Abrégé/Abstract:

The aim of the invention is to improve a method for encapsulating solder metal powders and the microencapsulated solder in such a way that the metal powder is reliably protected from external oxidising influences and the capsule only releases the metal powder as a result of the influence of temperature, without the influence of soldering flux. To this end, the method comprises the following steps: a) producing a suspension consisting of powder and a hydrophobic liquid; b) producing a hydrophobic surface layer on each metal particle by adding a cationic tenside; c) stirring the mixture obtained in steps a) and b) until a viscous, homogeneous mass is formed; d) mixing a radically polymerisable monomer into the mass obtained in step c); e) adding an organic initiator to start an interfacial polymerisation reaction; f) introducing the mass obtained in step e) into an aqueous substance, constantly stirring and controlling the polymerisation reaction by tempering to 50 to 90°C, and maintaining this temperature for at least 120 minutes and g) cooling, washing and separating the encapsulated solder powder.

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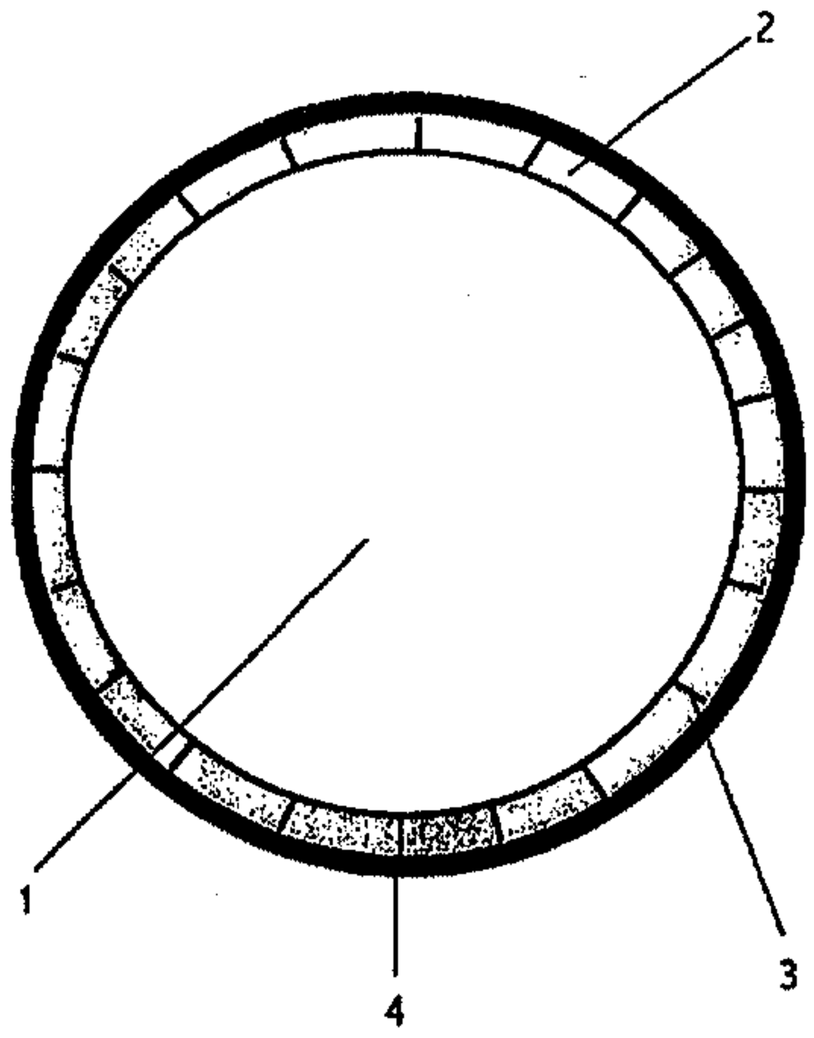
(54) Title: METHOD FOR ENCAPSULATING SOLDER METAL POWDERS AND SOLDER METAL POWDERS PRODUCED ACCORDING TO THIS METHOD

(54) Bezeichnung: VERFAHREN ZUM VERKAPSELN VON LOTMETALLPULVER UND DANACH HERGESTELLTE LOTMETALLPULVER

(57) Abstract

The aim of the invention is to improve a method for encapsulating solder metal powders and the microencapsulated solder in such a way that the metal powder is reliably protected from external oxidising influences and the capsule only releases the metal powder as a result of the influence of temperature, without the influence of soldering flux. To this end, the method comprises the following steps: a) producing a suspension consisting of powder and a hydrophobic liquid; b) producing a hydrophobic surface layer on each metal particle by adding a cationic tenside; c) stirring the mixture obtained in steps a) and b) until a viscous, homogeneous mass is formed; d) mixing a radically polymerisable monomer into the mass obtained in step c); e) adding an organic initiator to start an interfacial polymerisation reaction; f) introducing the mass obtained in step e) into an aqueous substance, constantly stirring and controlling the polymerisation reaction by tempering to 50 to 90°C, and maintaining this temperature for at least 120 minutes and g) cooling, washing and separating the encapsulated solder powder.



(57) Zusammenfassung

Der Erfindung liegt die Aufgabe zugrunde, das Verfahren zum Verkapseln von Lotmetallpulvern und das mikroverkapselte Lot derart zu verbessern, daß das Metallpulver sicher vor äußeren oxidativen Einwirkungen geschützt und gleichzeitig die Kapselung das Metallpulver ohne Flußmitteleinwirkung nur durch Temperatureinfluß frei gibt. Gelöst wird diese Aufgabe durch folgende Verfahrensschritte: a) Herstellen einer Suspension aus Pulver und einer hydrophoben Flüssigkeit, b) Erzeugen einer hydrophoben Oberflächenschicht auf jedem Metallpartikel durch Zusetzen eines kationischen Tensids, c) Rühren des Gemisches aus den Schritten a) und b) bis zur Bildung ener viskosen homogenen Masse, d) Zumischen eines radikalisch polymerisierbaren Monomers in die Masse des Schrittes c), e) Zugeben eines organischen Initiators zum Starten einer Grenzflächen-Polymerisationsreaktion, f) Einbringen der Masse des Schrittes e) in eine wäßrige Vorlage unter ständigem Rühren und Steuern der Polymerisationsreaktion durch Temperieren auf 50 bis 90 °C und Halten bei diesen Temperaturen für mindestens 120 min. und g) Abkühl en, Waschen und Abtrennen der gekapselten Lotpulver.

METHOD FOR ENCAPSULATING SOLDER METAL POWDERS AND SOLDER METAL POWDERS PRODUCED ACCORDING TO THIS METHOD

The invention relates to a method for encapsulating solder metal powders, in particular superfine solder metal powders, in which method the powders are provided with a thin polymer protective layer through a polymerization reaction running its course on the surface of the solder powder.

The invention further relates to a solder metal powder produced according to this method, with a core of solder metal with a diameter of 1 to 100 μm and a protective layer of polymer enclosing the core.

A method, belonging to the class, for producing micro-particulated reflow-solder agents is know from DE 44 02 042 A1, in which method the compact solder metal is melted down in a high-temperature organic liquid, with the aid of the stream-dispersion method is led to a sphero-symmetrical grain band of preferably 3 to 10 μm diameter, and the organic fluid is removed to the extent that the metal particulates remain covered, so that the fluid is to be converted into an emulsion and the individual particles of the suspension and emulsion are covered with a melamine polymerisate of the layer-thickness range of 50 to 250 nm according to the method of complex coacervation. The micro-particulated organic phase is subsequently quantitatively separated from the micro-particulated metal phase.

This micro-particulated metal powder is certainly protected by a thermosetting polymer system; however, it can only be released again through application of very strongly activated fluxing materials. These fluxing materials lead to a destruction of the microelectronic circuitry and are therefore unsuitable.

In addition, known from EP 0 556 864 A1 is a solder powder that is provided with a protective coating of parylenes. The parylene layer is applied by means of a vapor deposition, in which dimers are decomposed into monomers through pyrolysis, and these condense from the gas phase onto the surface of the powder particles, on which then the polymerization reaction and the film formation take place.

This known method has the disadvantage that the layer thickness required for use in solder pastes (<100 nm) does not guarantee complete encapsulation in the case of all of the particles and just as before a measurable permeability exists with respect to the small molecules damaging to solder powder, such as water and gases such as oxygen and sulfur dioxide.

Also, this known encapsulation material suffers the disadvantage that it can be dissolved only with highly active fluxing materials. Thus, an application in soldering processes has also not become known.

In light of this prior art, the object of the invention is to improve a method and micro-encapsulated solder so that the metal powder is reliably protected from outside oxidative effects, and at the same time the casing releases the metal powder without the actions of fluxing materials, merely through the influence of temperature.

This object is attained through the following method steps:

a) production of a suspension consisting of powder and a hydrophobic fluid;

b) generation of a hydrophobic surface layer on each metal particle through the addition of a cationic tenside with a chain length of C_1 to C_{20} under constant stirring in order to form a brush structure on the hydrophobic layer of step a);

c) stirring of the mixture from steps a) and b) until a viscous, homogeneous mass is formed;

d) admixture of a radically polymerizable monomer into the mass of step c), which monomer is capable of forming a thermoplastic polymer with a glazing temperature of T_g of at least 60°C below the solidification temperature of the solder powder;

e) addition of an organic initiator in order to start a boundary-surface polymerization reaction, integrating the hydrophobic layer of step b), and formation of a protective layer of thermoplastic polymer that has fluxing-material characteristics;

f) introduction of the mass of step e) into an aqueous receiver, the receiver containing an imulgator for suspension stabilization, and controlling of the polymerization reaction through temperatures of 50 to 90°C and maintaining of this temperature for at least 120 minutes;

g) cooling down, washing, and separation of the solder powder encapsulated in steps e) and f).

According to an additional preferred feature of the method according to the invention, used as hydrophobic fluids are non-drying vegetable and/or animal fats and/or aromatic solvents. Preferably, used as vegetable oils are castor oil, olive oil, or peanut oil.

In a further preferred development of the method according to the invention, used as tensides are quaternary ammonium salts, preferably dodecyltrimethyl ammonium chloride or cetyltrimethyl ammonium chloride.

A further preferred feature of the method according to the invention provides that methacrylic-acid-2-hydroxyethylester or methylmethacrylate is used as monomer.

For triggering the polymerization reaction, used as initiators are organic peroxides or azo compounds.

The object is further achieved with a solder metal powder by the fact that the metallic core forms, by means of a surfactant hydrophobic fluid and a tenside, a brush-like structure in which is mechanically anchored a thermoplastic, polymer capsule wall displaying flux-material characteristics, the capsule wall possessing a glazing temperature T_g of at least 60°C below the solidification temperature of the core.

The hydrophobic layer on the metal core, according to a further feature, consists of a non-drying vegetable and/or animal fat and/or aromatic solvents, preferably castor oil, olive oil, or peanut oil, and a cationic tenside with a chain length of C_1 to C_{20}, whose length influences the layer thickness of the casing wall. Appropriate monomers for the formation of the casing wall are radically polymerizable monomers, preferably methacrylic-acid-2-hydroxyethylester or methylmethacrylate.

Through the micro-encapsulation according to the invention, one thing achieved is a preservation of the powder characteristics. At the same time, the casing ensures the protection against an influence of atmospheric effects such as oxidation, atmospheric moisture, and formation of a covering layer. The oxidative attack on the metal particle inside the paste through activators is likewise prevented. By this means, the stability with respect to undesired chemical reactions with finished solder pastes is also increased. The use characteristics with respect to the workability and storage stability of the pastes are clearly improved.

The capsule wall, according to the invention, on these inorganic particles leads furthermore to a substantially improved coupling of these particles to the polymer matrix in applications such as pastes or pigments, which leads to a reduced tendency to sedimentation and better rheological characteristics.

The polymers used possess a glazing temperature, i.e. temperature at which the polymer converts into the plastic state, from 60 to 70°C. This ensures that the polymer capsule hull releases the metal powder before the melting down and the soldering process is not hindered. Through the choice of the monomer according to the invention, the capsule material preserves fluxing-material characteristics.

Further advantages and details result from the following description, with reference to the accompanying drawing.

In the following, the invention shall be explained in greater detail by way of several examples of embodiment.

The accompanying drawing shows the basic structure of a metal particle encapsulated according to the method according to the invention.

The metallic core has the reference numeral 1. Around the core 1 is placed a layer 2 of a non-drying, vegetable oil, for example castor oil, as a hydrophobic boundary surface. Extending from the core 1 is the longer-chain tenside 3, for example dodecyltrimethyl ammonium chloride, as a brush-like structure.

Locked together with this structure is the thermoplastic, polymer capsule wall 4.

Example 1:
For micro-encapsulation of the solder powder according to the invention, approximately 100 to 200 g of solder are weighed in with about 15% oil. Added to the weighed-in mass is a certain amount of a tenside solution. The amount of the tenside is here dependent on the type and, in the case of dodecyltrimethyl ammonium chloride, amounts to at least 0.053 mg per gram of solder. Conditional on the approximate share of oil in the weighed-in mass, an excess of tenside is possibly being worked with, which within certain limits is provably unproblematic for the further process (110% of the theoretical amount, relative to the entire weighed-in mass, shows no effects). After that, the solder-tenside-oil system is intensively mixed by stirring. When this mixture is homogeneous and a homogeneous mass forms, added to this are 1 to 4 mg of methacrylic-acid-2-hydroxyethylester per gram of solder and 0.1 to 0.3 mg of benzoyl peroxide per gram of monomer, and an intensive mixing takes place.

Placed in a glass beaker are 600–800 ml of water with an admixture of 0.1 to 1.0% polyvinyl pyrolidone K 90. This mixture is stirred by a dispersion apparatus, e.g. Ultra-Turrax of the fim IKA, at a rotational rate of 7200 rpm. To this is added the solder-oil-tenside mixture and the rotational rate is increased to 9000 rpm. After that, the system is brought to a temperature of 55°C. This temperature is maintained for 90 minutes and then increased to 65°C. The second reaction phase lasts 120 minutes. Subsequently, one allows the aqueous residue to cool to room temperature and decants the latter. The reaction vessel is rinsed with a small amount of ethanol, in order to make possible the transition to the final purification with dichloromethane.

After the alcohol is poured off, the solder powder located at the bottom of the glass beaker is stirred up in dichloromethane and transferred to another glass beaker. The "washing process" is repeated several times until the residue is pure. There follows the drying of the still-moist solder powder at room temperature.

Example 2:

250 g of solder powder are mixed with 14 g cetyltrimethyl ammonium chloride, 09 g methylmethacrylate, and 260 mg benzoyl peroxide in 40 g of peanut oil.

This reaction mixture is placed in 600 ml of water and suspended at 9000 rpm. As soon as a homogeneous suspension is attained, the temperature is brought to 60°C. After 90 minutes, the temperature is increased 10 degrees and this temperature is held constant for 120 minutes. After that, cooling off takes place, as well as further treatment corresponding to example 1.

Reference Numeral Table

metal core	1
oil layer	2
tenside	3
polymer capsule wall	4

CLAIMS:

1. Method for encapsulating solder metal powder in which the powder is provided with a thin protective polymer layer by a polymerization reaction running on the surface of the solder powder, with the following steps:

a) production of a suspension consisting of powder and a hydrophobic fluid;

b) generation of a hydrophobic surface layer on each metal particle through the addition of a cationic tenside with a chain length of C_1 to C_{20} under constant stirring in order to form a brush structure on the hydrophobic layer of step a);

c) stirring of the mixture from steps a) and b) until a viscous, homogeneous mass is formed;

d) admixture of a radically polymerizable monomer into the mass of step c), which monomer is capable of forming a thermoplastic polymer with a glass temperature of T_g of at least 60°C below the solidification temperature of the solder powder;

e) addition of an organic initiator in order to start a boundary-surface polymerization reaction, integrating the hydrophobic layer of step b), and formation of a protective layer of thermoplastic polymer that has fluxing-material characteristics;

f) introduction of the mass of step e) into an aqueous receiver, the receiver containing an imulgator for suspension stabilization, and controlling of the polymerization reaction through temperatures of 50 to 90°C and maintaining of this temperature for at least 120 minutes;

g) cooling down, washing, and separation of the solder powder encapsulated in steps e) and f).

2. Method according to claim 1, wherein the hydrophobic fluid is a non-drying vegetable or animal fat or an aromatic solvent.

3. Method according to claim 2, wherein the aromatic solvent is castor oil, olive oil or peanut oil.

4. Method according to claim 1, wherein the tenside is a quaternary ammonium salt.

5. Method according to claim 4, wherein the quaternary ammonium salt is dodecyltrimethyl ammonium chloride or cetyltrimethyl ammonium chlorite.

6. Method according to claim 1, wherein the monomer is methacrylic-acid-2-hydroxyethylester or methylmethacrylate.

7. Method according to claim 1, wherein the initiator is an organic peroxide or azo compound.

8. Micro-encapsulated solder metal powder for carrying out the method according to claim 1, with a core of solder metal with a diameter of 1 to 100 µm and a polymer layer enclosing the metal core, the core forming a brush-like structure with a surfactant hydrophobic fluid and a tenside, in which structure is mechanically anchored a thermoplastic polymer capsule wall displaying flux-material characteristics, and the polymer capsule wall possessing a glass temperature T_g of at least 60°C below the solidification temperature of the core.

9. Micro-encapsulated solder metal powder according to claim 8, wherein the hydrophobic fluid is a non-drying vegetable or animal fat or aromatic solvent.

10. Micro-encapsulated solder metal powder according to claim 9, wherein the aromatic solvent is castor oil, olive oil or peanut oil.

11. Micro-encapsulated solder metal powder according to claim 8, wherein the tenside is a cationic tenside with a chain length of C_1 to C_{20}, whose length influences the layer thickness of the polymer layer.

12. Micro-encapsulated solder metal powder according to claim 8, wherein the

polymer is formed from a radically polymerizable monomer.

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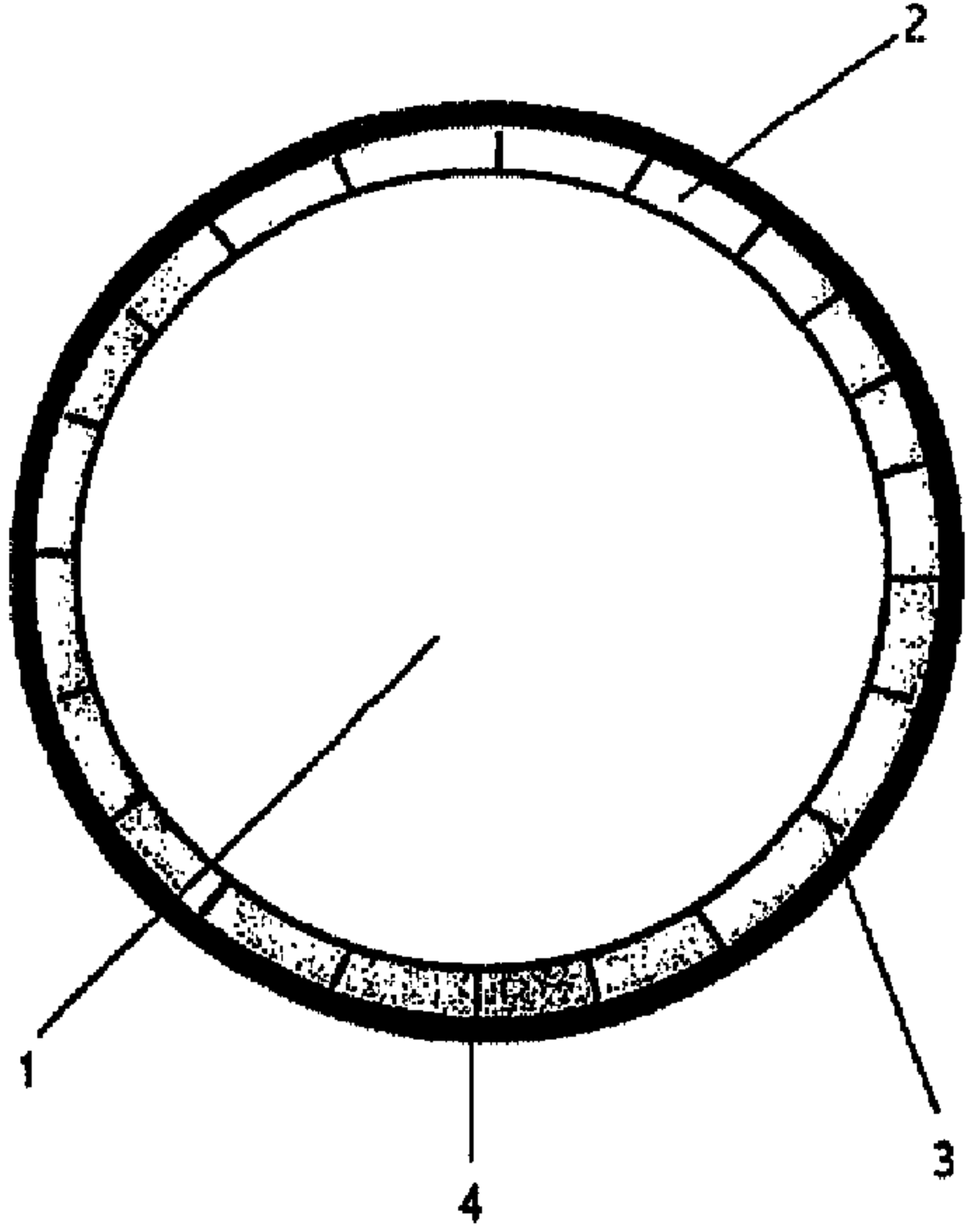
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