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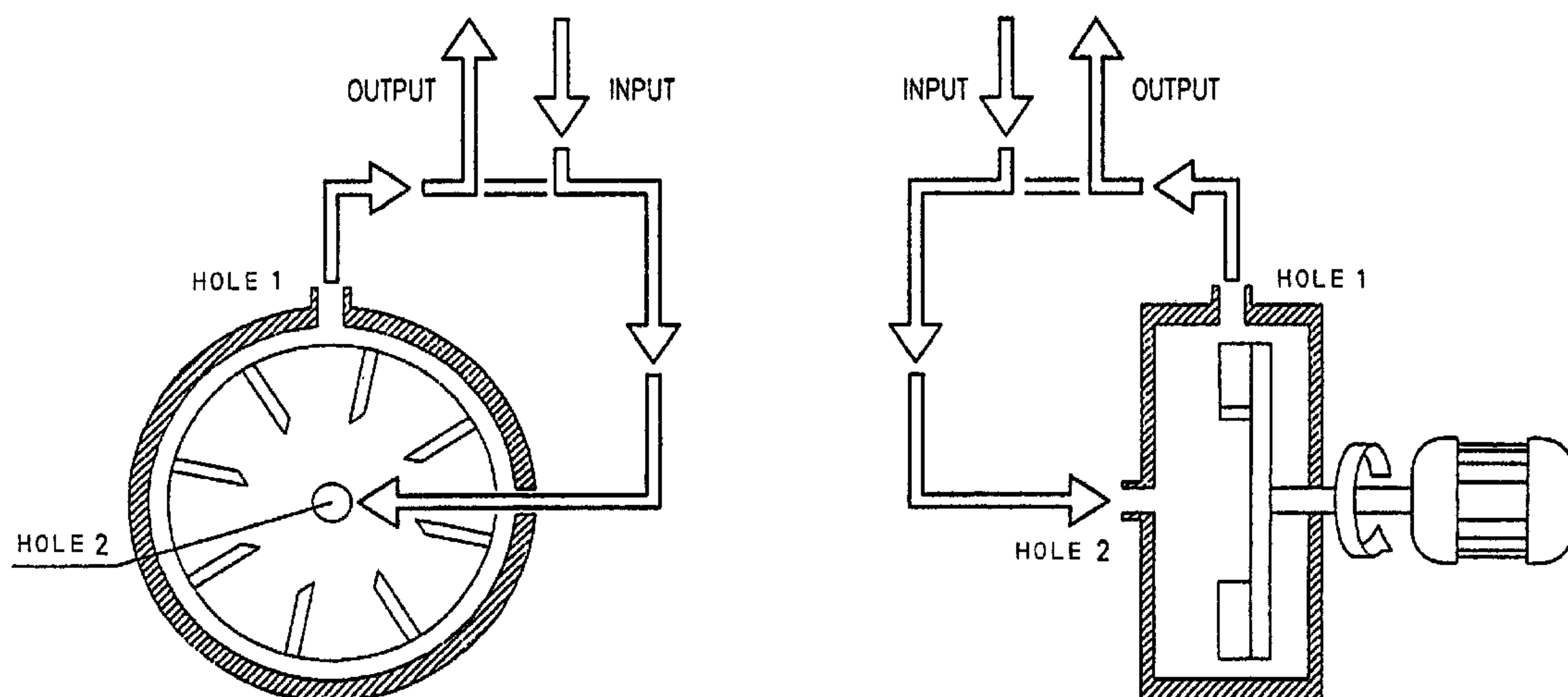
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(54) Titre : PROCEDE DE PRODUCTION DE POUDRE DE GRAPHITE A DENSITE APPARENTE AUGMENTEE

(54) Title: PROCESS FOR THE PRODUCTION OF GRAPHITE POWDERS OF INCREASED BULK DENSITY



(57) Abrégé/Abstract:

The invention relates to a method for increasing the Scott density of synthetic and/or natural graphite powders of any particle size distribution, preferably of highly-pure graphite, by subjecting the graphite powder to an autogenous surface treatment. The inventive powder is used, in particular, for producing dispersions, coatings with an increased graphite/binder ratio and increased electric and thermal conductivity, gas and liquid-tight coatings on metal substrates, thermoplastic or duroplastic graphite-polymer composites, or for producing metallic, non-ferrous sintering materials.



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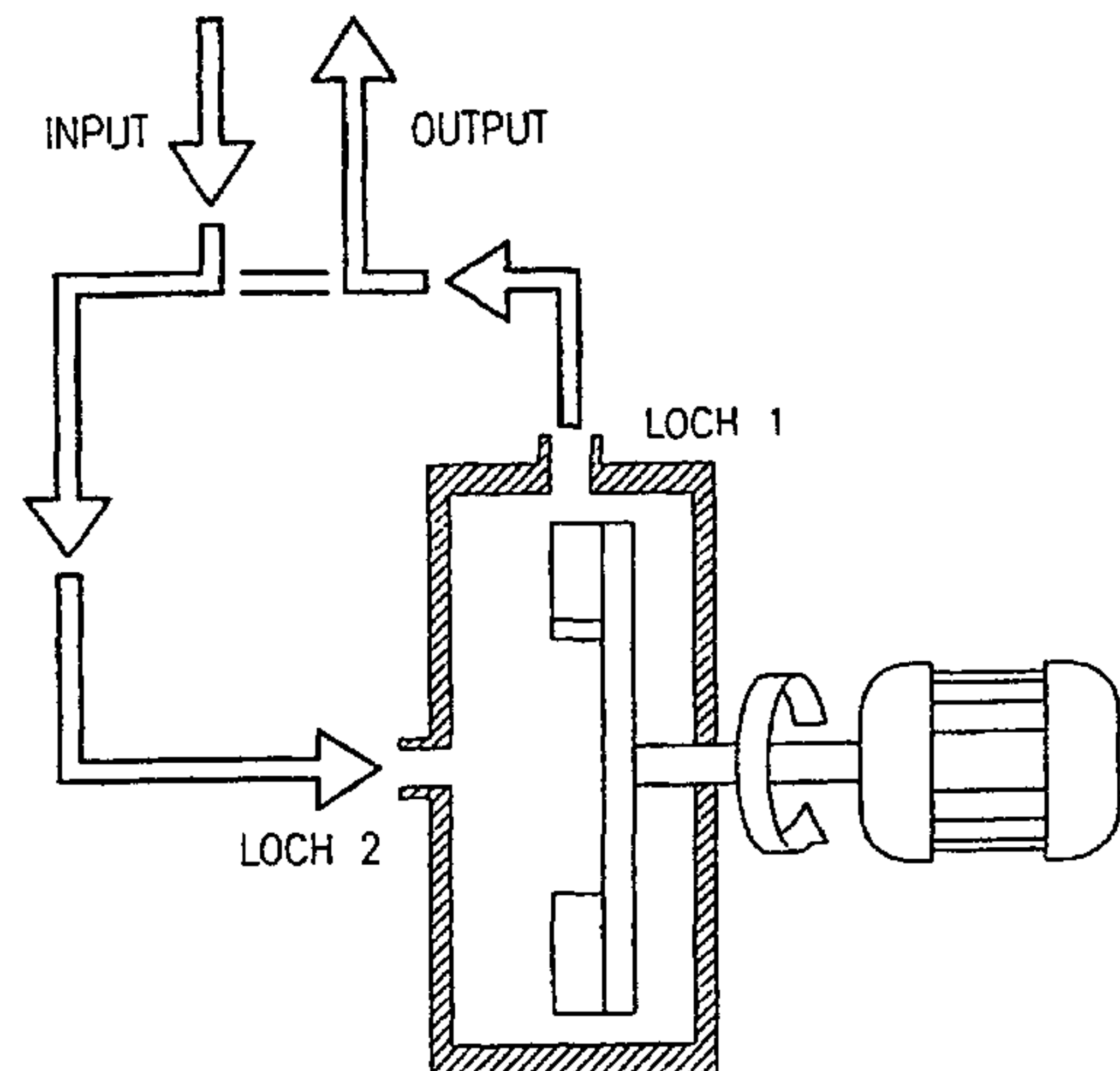
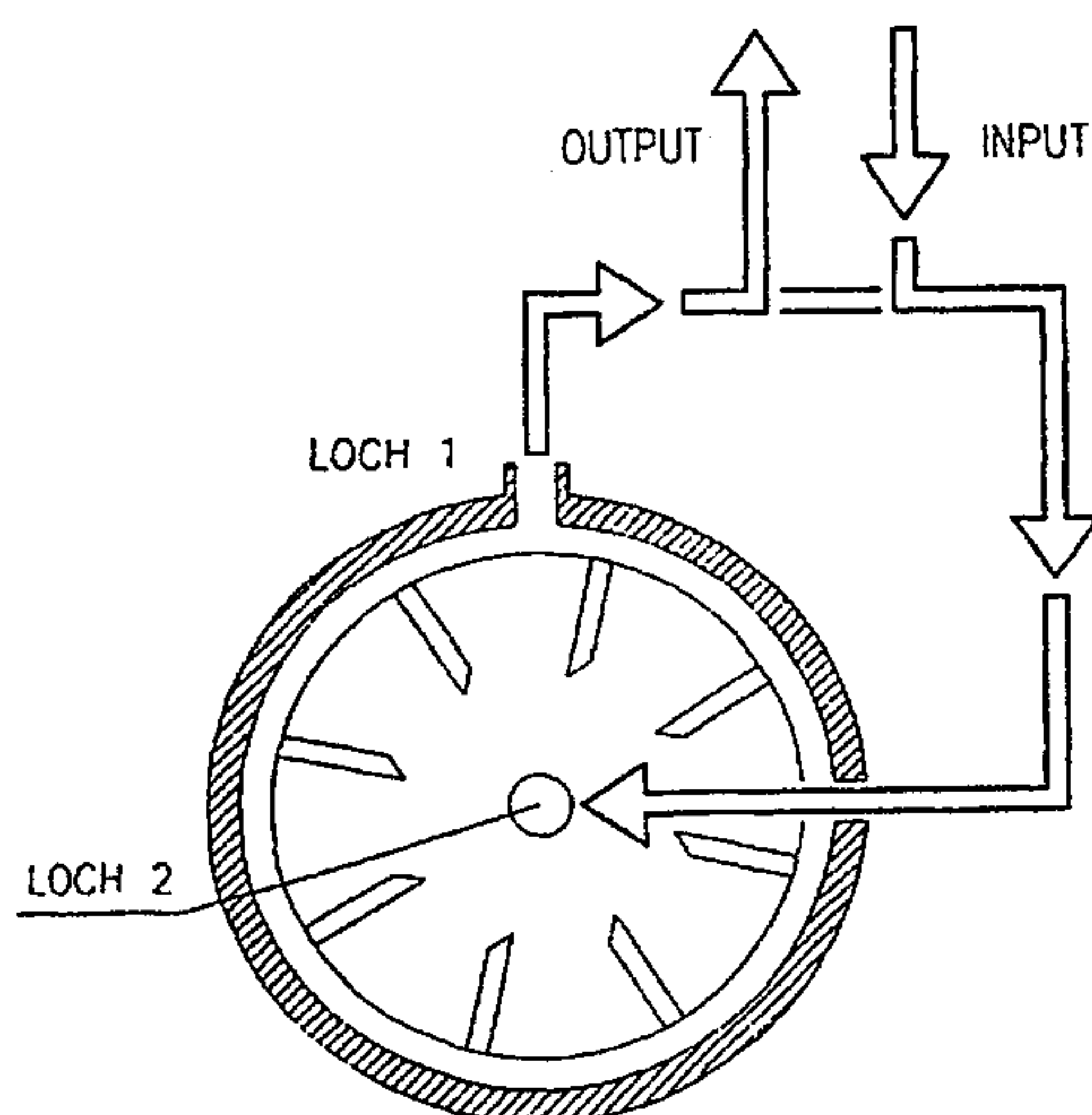
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(54) Title: METHOD FOR PRODUCING GRAPHITE POWDER WITH AN INCREASED BULK DENSITY

(54) Bezeichnung: VERFAHREN ZUR HERSTELLUNG VON GRAPHITPULVERN MIT ERHÖHTER SCHÜTTDICHTHE



(57) Abstract: The invention relates to a method for increasing the Scott density of synthetic and/or natural graphite powders of any particle size distribution, preferably of highly-pure graphite, by subjecting the graphite powder to an autogenous surface treatment. The inventive powder is used, in particular, for producing dispersions, coatings with an increased graphite/binder ratio and increased electric and thermal conductivity, gas and liquid-tight coatings on metal substrates, thermoplastic or duroplastic graphite-polymer composites, or for producing metallic, non-ferrous sintering materials.

(57) Zusammenfassung: Verfahren zur Erhöhung der Scott-Dichte von synthetischen und/oder natürlichen graphitischen Pulvern beliebiger Partikelgrößenverteilung, vorzugsweise von hochreinem Graphit, indem man das graphitische Pulver einer autogenen Oberflächenbehandlung unterzieht. Verwendung der erfindungsgemässen Pulver insbesondere zur Herstellung von Dispersionen, von Beschichtungen mit erhöhtem Graphit/Binder-Verhältnis und erhöhter elektrischer und thermischer Leitfähigkeit, von gasdichten und flüssigkeitsdichten Beschichtungen auf Metallsubstraten, von thermoplastischen oder duroplastischen Graphit-Polymerkompositen oder von metallischen Nichteisen-Sintermaterialien.

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Zur Erklärung der Zweibuchstaben-Codes, und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

Process for the production of graphite powders of increased bulk density

The present invention relates to a process for the production of graphite powders of increased bulk density. The present invention relates in particular to an autogenous surface treatment of any pulverulent graphitic materials, their bulk density and tamped density being markedly increased and other important material properties being advantageously modified as a result of the mutual physical-mechanical action of the individual powder particles.

Graphitic materials, especially those with a high graphite content, are known per se and are used in industry in a variety of ways. High-purity graphitic carbons have xylene densities (also called single-crystal densities or real densities) ranging from 1.80 to 2.27 g.cm⁻³ and a crystal structure which can be characterized by a c/2 value of 0.3354 to 0.3360 nm and an L_c value of more than 40 nm (L_c > 40 nm). These materials are obtained from natural sources, enriched and purified or produced synthetically from amorphous carbon products in a high temperature process. Subsequent grinding processes produce pulverulent materials with different mean particle sizes in each case. A given particle size for a powder is normally always a mean value of a specific particle size distribution. The particle size distribution to be used for a particular purpose depends especially on the composition of the graphitic material and the associated properties, as well as on the intended use.

The particle shape is always platelet-like, the anisotropy of the particles being the more pronounced the higher the xylene density and L_c values. The Scott density (also referred to as bulk density) of such materials, for example with particle sizes smaller than 100 micron (particle size < 100 µm, determined by laser diffraction analysis), is normally below 0.25 g.cm⁻³, the Scott density being the lower the smaller the particle size. Comminution of the particles by grinding generally results in a lowering of the Scott density. The Scott density can be somewhat increased by an optimized particle size distribution. Thus, for example, Scott densities up to max. 0.3 g.cm⁻³ are achieved by an optimized composition of fine and coarse fractions for such materials with particle sizes below 100 micron.

The tamped density, the compressibility and the absorption capacity for polymeric

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binder materials and liquids such as oils, and for organic solvents and aqueous systems, are equally important properties of graphite powders. These properties correlate with the composition of the graphite powders and especially with the particle size distribution.

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It has now been found that, surprisingly, the values of the Scott density for a particular graphite powder of any particle size distribution is considerably increased when the graphite powder is subjected to an autogenous surface treatment in which the particles impact with one another at an appropriate speed and for a sufficient length of time. The impacts and the associated mutual physical-mechanical action change the structure or surface of the graphite particle in such a way as to result in a considerable increase in the Scott density. The other properties mentioned above are also modified to a considerable extent.

10

Under the electron microscope, the crude, ground, platelet-like graphite particle has an irregular shape and sharp edges. The irregular particle contours are abraded and the edges rounded off by the treatment according to the invention. If the energy dose is appropriately optimized, the grinding effect which occurs with other mechanical treatments, leading to a noticeable lowering of the bulk density, is considerably reduced or minimized. Although the abrasion of the particles creates dust, which, together with a minimal grinding effect, leads to a slight reduction in particle size and Scott density (bulk density), this particle size effect is far outweighed by the surprisingly large total increase in Scott density, and the change in the other properties, caused by the treatment according to the invention. The present invention can be at least partly explained by the observed changes in the particle contours, but the invention is not bound to this explanation.

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The present invention is defined in the Claims. In particular, the present invention relates to a process for increasing the Scott density of graphite powders of any particle size distribution, characterized in that the graphite powder is subjected to an autogenous surface treatment.

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The process of autogenous surface treatment consists in allowing the individual powder particles to impact with one another at a measured speed so that, as a result

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of the associated mutual physical-mechanical action of the individual particles, their surface structure changes but the individual particle remains substantially unbroken, i.e. no substantial grinding effect occurs. This change in the particle contour or surface structure of the individual particle gives rise to the increase in Scott density according to the invention. The autogenous surface treatment is carried out, and the individual particles are allowed to act on one another, until the desired Scott density is achieved. The measured speed means that the speed or energy with which the individual particles are charged is adjusted so that the particles do not disintegrate on impact or collision, thereby practically avoiding a grinding effect. This adjustment is a question of process optimization and does not present a problem to those skilled in the art.

The Scott density achievable by means of the optimized grinding effect for a graphite powder of any particle size distribution can be increased in each case by at least about 10% to about 100%, preferably by about 20% to 80%, by the autogenous surface treatment according to the invention. Hitherto unattained Scott densities of 0.45 g/cm³ or more are thus achieved for graphitic materials.

The tamped density achievable by means of the optimized grinding effect for a graphite powder of any particle size distribution can also be increased by at least about 10% to 100%, preferably by about 20% to 80%, by the process according to the invention. Hitherto unattained tamped densities of at least 0.90 g/cm³ are thus achieved for graphite powders.

In the case of particle sizes of <100 µm, the autogenous surface treatment according to the invention is preferably carried out by fluidizing or dispersing the graphite powder particles in an inert carrier gas and accelerating the particles with the aid of the carrier gas, as described below. The intensity of this treatment is determined by the carbon type and the mass of the particles, their speed and the amount of material used per treatment, i.e. the concentration of the fluidized particles dispersed in the gas. The intensity of the treatment increases with the softness of the graphitic carbon used, the mass of the particles, their speed and the amount used. For particle sizes of <300 µm, the dispersion and acceleration of the particles are preferably effected by means of rotating mechanical tools, for example

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in the present process by means of a turbine or directly by means of a rotating disk.

However, the grinding effect which occurs also increases simultaneously with increasing intensity of the treatment. Thus, to achieve the maximum bulk density of a material, there is a maximum intensity which results from the optimized parameters of particle speed, particle mass and amount used. The formation of agglomerates due to the agglutination of smaller particles, which would also lead to a sustained increase in the Scott density, has not been observed. Treated particles larger than the untreated particles used did not appear in any of the experiments performed. Analyses of the treated materials by scanning electron microscopy also showed no such agglomeration.

The treatment according to the invention not only increases the Scott density but also improves the compressibility properties of the graphite powders and reduces their absorption capacity for polymeric binder materials and liquids such as oils, organic solvents and aqueous systems. The crystallinity of the graphitic carbon particles, on the other hand, remains unaffected by the mechanical surface treatment. The structural parameters and the xylene density also remain unchanged compared with the untreated particles.

The process according to the invention also increases the pressed density achievable by the optimized grinding effect for a graphite powder of any particle size distribution by at least about 0.5% to 10%, preferably by about 1% to 8%. If the powders treated according to the invention are used to produce mouldings by compression under a pressure of 2.5 to/cm², markedly higher pressed densities can be achieved compared with the untreated materials.

Furthermore, the powders treated according to the invention exhibit a markedly reduced oil absorption capacity and binder uptake ranging from about 10% to 50% and especially from an average of about 20% to 45%, values in excess of 50% also being obtainable. This effect is achieved by the treatment according to the invention because the porosity (pore structure) of the particles is not affected by the treatment, as can be demonstrated by the fact that the nitrogen adsorption properties and xylene densities hardly change.

Said markedly reduced absorption properties also result in markedly lower viscosities of dispersions of the graphite powders treated according to the invention in liquid media, so dispersions with a correspondingly increased solids content can be prepared with the graphite powders treated according to the invention. The solids content of liquid carbon dispersions can be increased by more than 5% to over 30% by using graphite powders treated according to the invention.

Graphite powders suitable for the use according to the invention are especially those with a high graphite content in the particle, and particularly so-called high-purity graphites, preferably with xylene densities ranging from 1.80 to 2.27 g.cm⁻³ and a crystal structure characterized by a c/2 value of 0.3354 to 0.3360 nm and an L_c value of more than 40 nm (L_c > 40 nm). The powders can be obtained from natural sources or prepared synthetically from amorphous carbon products and can have any mean particle size and particle size distribution. Preferred pulverulent graphitic materials are those with a mean particle size of up to 150 µm, preferably of 1 µm to 50 µm, and especially high-purity pulverulent graphites. Such graphites are known per se.

The process according to the invention is preferably carried out in such a way that the graphite powder particles to be treated are dispersed and fluidized in a gas. This can be done using any method of fluidization technology known per se in which the particles impact with one another in the fluidized state and thereby change their surface contours and surface structures, as is the case e.g. in a fluidized bed. However, to carry out the process according to the invention, the fluidized particles are preferably provided with higher speeds so that the particles fluidized in this way are accelerated with higher energies. Preferably, the fluidized particles are continuously concentrated and diluted again in the gaseous environment. The resulting collisions between the particles set in rotation, and the friction between them, result in surface abrasion of the particles, the energy transferred to the particles being adjusted so that the collisions and friction cause substantially no disintegration of the particles.

The process according to the invention can be put into optimum effect e.g. in the device shown in Figure 1. This device consists specifically of a circular disk with

radial impact pins flush-mounted on the rim, said disk being sheathed by a cylindrical treatment chamber closed to the outside (turbine with associated turbine effect). The dimensions of the cylindrical treatment chamber are adjusted so that it encloses the disk and can allow some space between its inner wall and the rotating disk. The disk is connected to a motor, located outside the treatment chamber, by means of a shaft through the wall of the treatment chamber and can be set in rotation by this motor. The cylindrical treatment chamber is provided with a radial aperture (hole). An additional aperture is provided in the cylinder jacket of the treatment chamber, perpendicular to the disk and disk axis. These two apertures are connected by a tube located outside the treatment chamber. Thus a tube running outside the treatment chamber and attached to the wall of the treatment chamber connects the periphery of the treatment chamber to its centre. The gas (fluid) containing the fluidized particles, accelerated centrifugally by the rotating disk, circulates through this external treatment tube, exiting through the tube at the periphery of the treatment chamber as a result of the centrifugal force and flowing back through the other end of this tube into the centre of the treatment chamber, where it is accelerated again. The particles of material are accelerated by the impact pins of the rotating disk and driven away in a peripheral direction by the centrifugal forces produced by the high-speed rotor. The particles dispersed and accelerated into the gas in this way circulate in the machine along the inside of the cylinder jacket. The particles reaching the inlet of the circulation tube enter the tube and return to the treatment chamber in the region of the centre of the machine. This results in a continuous concentration and dilution of the particles in the surrounding gaseous medium. A fraction of the treated particles is continuously fed into or withdrawn from an attached tube, but the process can also be carried out as a batch process.

The graphite powders treated according to the invention can advantageously be used as pigments in aqueous or solvent-based dispersions, thereby achieving higher solids contents than with untreated powders. The viscosity of liquid dispersions of materials treated according to the invention is markedly lower for the same solids content. Also, when dispersions according to the invention are applied to substrates and dried, coatings with markedly lower porosity values are obtained because the content of liquid phase is markedly lower. The higher solids content

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also means that smaller binder/carbon ratios are needed to stabilize a dried carbon coating on a substrate. The low polymeric binder contents result in a marked increase in the electrical and thermal conductivities of such carbon layers.

- 5 Dispersions containing mixtures of synthetic and/or natural graphitic carbons treated according to the invention and a polymeric binder in an aqueous or solvent-based medium can be applied to metal foils and dried to give stable coatings (for thicknesses of 10 to 5000 μm) with an increased graphite/binder ratio and hence also increased electrical and thermal conductivities. The porosities of the dried
- 10 films are normally below 50% and are thus appreciably lower than those of films formed of conventional graphites. Such dispersions can therefore advantageously also be used for gas-tight and liquid-tight coatings on metal substrates, which can be used as electrically conducting anticorrosive films on metal foils and plates.
- 15 The dried coatings formed by the graphites treated according to the invention can be compressed by a calender without the graphite film delaminating from the metal foil. This delamination from the metal foil is frequently observed with untreated graphites. The calendering of graphite films produced from graphite powders treated according to the invention affords coatings with porosities below 30%
- 20 without altering the texture or particle structure of the graphite powders used. Such film coatings on metal foils, characterized by porosities below 30% and stabilized with lower binder/carbon ratios, can be used in lithium ion batteries as negative electrodes with charge densities above 550 Ah/l. The current-carrying capacity of such electrodes is markedly higher than that of electrodes made of
- 25 conventional graphite powders. Such negative electrodes can thus be used very advantageously for lithium ion cells with a high power density.

- The high packing density of the synthetic or natural graphites treated according to the invention, combined with the relatively low polymeric binder absorption
- 30 capacity, is advantageous in the production of graphite/polymer composites which can be compressed to gas-tight graphite plates of high electrical conductivity. Such plates are advantageously used as bipolar plates in polymer electrolyte fuel cell technology.

5 Mixtures of polymers with synthetic or natural graphites or graphitic carbons
treated according to the invention form thermoplastic or thermosetting composites
with a higher proportion of carbon filler and a lower processing viscosity.
Thermoplastic polymer/graphite composite materials with graphites treated
according to the invention have higher (and hence improved) values in respect of
their isotropic, mechanical, thermal and electrical properties and behave more
10 isotropically than composites with untreated graphitic carbons.

Metallic non-ferrous sintered materials which have been produced with synthetic
or natural graphitic carbons treated according to the invention, or contain such
carbons, have improved isotropic, mechanical and tribological properties.

15

The Examples which follow describe the invention.

Examples 1 to 5 show the material properties of various graphites before and after
the autogenous surface treatment according to the invention. The experiments
20 were performed in the device described in the above section. The rotating disk
used had a periphery of 0.75 m and a speed of rotation of 4800 rpm.

Examples 1 - 5 were carried out under the experimental conditions given in Table
1.

Table 1

Example	Type of graphite	Amount used	Treatment time	Speed of rotating disk
1	TIMREX® KS-graphite	150 g	5 min	4800 rpm
2	TIMREX® SLX-graphite	150 g	5 min	4200 rpm
3	TIMREX® SLM-graphite	150 g	5 min	4800 rpm
4	TIMREX® SFG-graphite	200 g	5 min	4800 rpm
5	TIMREX® NP-graphite	200 g	7 min	4800 rpm
6	TIMREX® KS 5-75 TT	200 g	5 min	4800 rpm

TIMREX® KS-graphite = TIMREX® KS 5-25 from TIMCAL AG

TIMREX® SLX-graphite = TIMREX® SLX 50 from TIMCAL AG

5 TIMREX® SLM-graphite = TIMREX® SLM 44 from TIMCAL AG

TIMREX® SFG-graphite = TIMREX® SFG 44 from TIMCAL AG

TIMREX® NP-graphite = TIMREX® NP 44 from TIMCAL AG

Examples 1 to 5 show a marked increase in Scott density (bulk density) and
10 tamped density for the powders treated according to the invention. The treated
powders exhibited no agglomerates whatsoever. The resulting change in particle
size distribution is indicative of a small grinding effect. The slight lowering of d
values, however, is caused especially by the dust produced by the abrasion of the
particles. The pore structure of the treated particles is not affected by the surface
15 treatment. It is assumed that the dust produced by the treatment and the slight
decrease in particle size distribution are the main reason for the slight lowering of
the L_c values and the xylene densities. The elastic recovery of the compressed
treated materials drops sharply. The pressed density of mouldings produced from
the treated materials under a pressure of 2.5 to/cm² increases sharply. Although the
20 BET values are increased somewhat, the oil absorption and binder absorption of
the particles treated according to the invention decrease markedly. Dispersions of
treated carbon particles in liquid media exhibit markedly lower viscosities than
dispersions of untreated carbon particles. The solids content of liquid carbon

dispersions can be increased by more than 5% by using carbon particles according to the invention. The electrical resistance of the carbons treated according to the invention increases. The changes in surface contours of the individual particles which result from the treatment of powders according to the invention can be
5 clearly seen from scanning electron micrographs.

Experimental section

The particle size distribution of the materials was determined by laser diffraction analysis using a MALVERN Mastersizer. The structural parameters were obtained
10 from X-ray diffraction experiments based on the $\text{CuK}_{\alpha 1}$ line. The crystallographic cell constant in the c direction ($c/2$) was determined from the relative position of the (002) or (004) diffraction reflex. The maximum height of the single-crystal domains in a particle in the crystallographic c direction, L_c , and the resulting number of ideally stacked graphite planes were obtained from the (002) or (004)
15 diffraction reflex according to the model of Scherrer and Jones (P. Scherrer, Göttinger Nachrichten 2 (1918) p. 98; F.W. Jones, Proc. Roy. Soc. (London) 166 A (1938) p. 16). The xylene density was determined according to DIN 51 901. Determination of the Scott density was based on ASTM B 329. The tamped density was determined according to AKK-19. The specific surface areas were
20 determined by the method of Brunauer, Emmett and Teller using a Micromeritics ASAP 2010. To determine the elastic recovery, the material was placed under a pressure of 0.5 to/cm². The recovery was obtained from the height of the moulding with and without applied pressure and is given in percent. The electrical resistance was measured according to DIN 51 911 using a moulding produced under a
25 pressure of 2.5 to/cm². The pressed density of this moulding is also given. The oil absorption was measured on the basis of DIN ISO 787 with initial weights of 0.5 g of material and 1.5 g of oil. The mixture was centrifuged in a Sigma 6-10 centrifuge for 90 min at a speed of 1500 rpm.

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Example 1

TIMREX® KS synthetic graphite Untreated	TIMREX® KS synthetic graphite After treatment
Particle size $d_{10} = 7.0$ micron $d_{50} = 15.2$ micron $d_{90} = 30.2$ micron	Particle size $d_{10} = 5.9$ micron $d_{50} = 13.5$ micron $d_{90} = 27.4$ micron
$L_c(002)/L_c(004)$ 120 nm/68 nm	$L_c(002)/L_c(004)$ 101 nm/64 nm
$c/2(002)/c/2(004)$ 0.3355 nm/0.3355 nm	$c/2(002)/c/2(004)$ 0.3355 nm/0.3355 nm
Xylene density 2.254 g.cm^{-3}	Xylene density 2.248 g.cm^{-3}
Scott density 0.23 g.cm^{-3}	Scott density 0.30 g.cm^{-3}
Tamped density 0.539 g.cm^{-3}	Tamped density 0.674 g.cm^{-3}
BET specific surface area $8.6 \text{ m}^2.\text{g}^{-1}$	BET specific surface area $9.3 \text{ m}^2.\text{g}^{-1}$
Elastic recovery 17%	Elastic recovery 12.3%
Electrical resistance 1.911 mΩ.cm	Electrical resistance 2.085 mΩ.cm
Oil absorption $113.5\% \pm 1.3\%$	Oil absorption $64.3\% \pm 0.2\%$
Pressed density (2.5 to/cm ²) 1.863 g.cm^{-3}	Pressed density (2.5 to/cm ²) 1.957 g.cm^{-3}

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Example 2

TIMREX® SLX synthetic graphite Untreated	TIMREX® SLX synthetic graphite After treatment
Particle size $d_{10} = 11.6$ micron $d_{50} = 27.3$ micron $d_{90} = 52.5$ micron	Particle size $d_{10} = 7.4$ micron $d_{50} = 20.4$ micron $d_{90} = 40.8$ micron
$L_c(002)/L_c(004)$ >500 nm/232 nm	$L_c(002)/L_c(004)$ 368 nm/158 nm
$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm	$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm
Xylene density 2.261 g.cm^{-3}	Xylene density 2.258 g.cm^{-3}
Scott density 0.30 g.cm^{-3}	Scott density 0.38 g.cm^{-3}
Tamped density 0.641 g.cm^{-3}	Tamped density 0.778 g.cm^{-3}
BET specific surface area $4.0 \text{ m}^2.\text{g}^{-1}$	BET specific surface area $5.9 \text{ m}^2.\text{g}^{-1}$
Elastic recovery 7.7%	Elastic recovery 4.6%
Electrical resistance 0.986 m Ω .cm	Electrical resistance 1.166 m Ω .cm
Oil absorption $94.7\% \pm 11.9\%$	Oil absorption $73.3\% \pm 1.9\%$
Pressed density (2.5 to/cm ²) 2.036 g.cm^{-3}	Pressed density (2.5 to/cm ²) 2.051 g.cm^{-3}

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Example 3

TIMREX® SLM synthetic graphite Untreated	TIMREX® SLM synthetic graphite After treatment
Particle size $d_{10} = 7.3$ micron $d_{50} = 23.2$ micron $d_{90} = 49.4$ micron	Particle size $d_{10} = 4.3$ micron $d_{50} = 13.9$ micron $d_{90} = 35.0$ micron
$L_c(002)/L_c(004)$ 241 nm/139 nm	$L_c(002)/L_c(004)$ 196 nm/116 nm
$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm	$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm
Xylene density 2.254 g.cm^{-3}	Xylene density 2.252 g.cm^{-3}
Scott density 0.19 g.cm^{-3}	Scott density 0.34 g.cm^{-3}
Tamped density 0.408 g.cm^{-3}	Tamped density 0.738 g.cm^{-3}
BET specific surface area $4.9 \text{ m}^2.\text{g}^{-1}$	BET specific surface area $7.7 \text{ m}^2.\text{g}^{-1}$
Elastic recovery 14.0%	Elastic recovery 8.6%
Electrical resistance 1.278 m Ω .cm	Electrical resistance 1.741 m Ω .cm
Oil absorption $109.5\% \pm 2.7\%$	Oil absorption $75.0\% \pm 5.3\%$
Pressed density (2.5 to/cm ²) 1.930 g.cm^{-3}	Pressed density (2.5 to/cm ²) 2.036 g.cm^{-3}

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Example 4

TIMREX® SFG synthetic graphite Untreated	TIMREX® SFG synthetic graphite After treatment
Particle size $d_{10} = 7.5$ micron $d_{50} = 24.1$ micron $d_{90} = 49.2$ micron	Particle size $d_{10} = 4.4$ micron $d_{50} = 15.0$ micron $d_{90} = 35.5$ micron
$L_c(002)/L_c(004)$ 320 nm/138 nm	$L_c(002)/L_c(004)$ 283 nm/199 nm
$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm	$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm
Xylene density 2.262 g.cm^{-3}	Xylene density 2.258 g.cm^{-3}
Scott density 0.20 g.cm^{-3}	Scott density 0.36 g.cm^{-3}
Tamped density 0.420 g.cm^{-3}	Tamped density 0.766 g.cm^{-3}
BET specific surface area $5.9 \text{ m}^2.\text{g}^{-1}$	BET specific surface area $7.4 \text{ m}^2.\text{g}^{-1}$
Elastic recovery 9.2%	Elastic recovery 5.6%
Electrical resistance 0.925 m Ω .cm	Electrical resistance 0.986 m Ω .cm
Oil absorption $110.2\% \pm 6.4\%$	Oil absorption $81.8\% \pm 6.9\%$
Pressed density (2.5 to/cm ²) 2.005 g.cm^{-3}	Pressed density (2.5 to/cm ²) 2.036 g.cm^{-3}

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Example 5

TIMREX® NP purified natural graphite Untreated	TIMREX® NP purified natural graphite After treatment
Particle size $d_{10} = 6.6$ micron $d_{50} = 23.0$ micron $d_{90} = 49.5$ micron	Particle size $d_{10} = 3.7$ micron $d_{50} = 13.8$ micron $d_{90} = 36.9$ micron
$L_c(002)/L_c(004)$ 364 nm/166 nm	$L_c(002)/L_c(004)$ 255 nm/103 nm
$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm	$c/2(002)/c/2(004)$ 0.3354 nm/0.3354 nm
Xylene density 2.263 g.cm^{-3}	Xylene density 2.258 g.cm^{-3}
Scott density 0.24 g.cm^{-3}	Scott density 0.42 g.cm^{-3}
Tamped density 0.495 g.cm^{-3}	Tamped density 0.862 g.cm^{-3}
BET specific surface area $5.0 \text{ m}^2.\text{g}^{-1}$	BET specific surface area $7.9 \text{ m}^2.\text{g}^{-1}$
Elastic recovery 4.9%	Elastic recovery 3.8%
Electrical resistance 0.910 m Ω .cm	Electrical resistance 1.359 m Ω .cm
Oil absorption $107.2\% \pm 3.6\%$	Oil absorption $58.9\% \pm 0.6\%$
Pressed density (2.5 to/cm ²) 2.066 g.cm^{-3}	Pressed density (2.5 to/cm ²) 2.064 g.cm^{-3}

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Example 6

TIMREX® KS purified natural graphite Untreated	TIMREX® KS purified natural graphite After treatment
Particle size $d_{10} = 8.3$ micron $d_{50} = 38.4$ micron $d_{90} = 68.4$ micron	Particle size $d_{10} = 3.1$ micron $d_{50} = 38.4$ micron $d_{90} = 68.4$ micron
$L_c(002)/L_c(004)$ 142 nm/62 nm	$L_c(002)/L_c(004)$ 105 nm/52 nm
$c/2(002)/c/2(004)$ 0.3355 nm/0.3355 nm	$c/2(002)/c/2(004)$ 0.3356 nm/0.3356 nm
Xylene density 2.227 g.cm^{-3}	Xylene density 2.225 g.cm^{-3}
Scott density 0.44 g.cm^{-3}	Scott density 0.46 g.cm^{-3}
Tamped density 0.84 g.cm^{-3}	Tamped density 0.902 g.cm^{-3}
BET specific surface area $4.1 \text{ m}^2.\text{g}^{-1}$	BET specific surface area $8.0 \text{ m}^2.\text{g}^{-1}$
Elastic recovery 25%	Elastic recovery 14.68%
Electrical resistance $2.109 \text{ m}\Omega.\text{cm}$	Electrical resistance $2.311 \text{ m}\Omega.\text{cm}$
Oil absorption $97.2\% \pm 1.6\%$	Oil absorption $54.7\% \pm 0.8\%$
Pressed density (2.5 to/cm^2) 1.912 g.cm^{-3}	Pressed density (2.5 to/cm^2) 1.972 g.cm^{-3}

Claims

1. Process for increasing the Scott density of graphite powders of any particle size distribution, the graphite powder being a synthetic and/or natural graphitic carbon which has a high graphite content in the particle, characterized in that the graphite powder is subjected to an autogenous surface treatment in which that the individual powder particles are allowed to impact with one another at a measured speed so that their surface structure changes without a substantial grinding effect occurring.
2. Process according to Claim 1, characterized in that the graphite powder is high-purity graphite.
3. Process according to Claim 1 or 2, characterized in that the graphite powder has a xylene density ranging from 1.80 to 2.27 g.cm⁻³ and a crystal structure characterized by a c/2 value of 0.3354 to 0.3360 nm and an L_c value of more than 40 nm (L_c > 40 nm).
4. Process according to one of Claims 1 - 3, characterized in that the graphite powder has a particle size of up to 150 µm, preferably of 1 µm to 50 µm.
5. Process according to one of Claims 1 - 4, characterized in that the autogenous surface treatment is carried out until the Scott density and/or the tamped density of the starting powder has increased by at least about 10% to about 100%, preferably by about 20% to 80%.
6. Process according to Claim 5, characterized in that the autogenous surface treatment is carried out by fluidizing or dispersing graphite powder particles with sizes of <100 µm in an inert carrier gas with the aid of the carrier gas.
7. Process according to Claim 5, characterized in that the autogenous surface treatment is carried out by dispersing graphite powder particles with sizes of <300 µm by means of a rotating mechanical tool, preferably by means of

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a turbine.

8. Use of the graphite powders produced according to one of Claims 1 - 7 for the preparation of dispersions in liquid media with an increased solids content.
9. Use of the graphite powders produced according to one of Claims 1 - 7 as pigments in aqueous or solvent-based dispersions.
10. Use of dispersions containing mixtures of graphitic carbons according to one of Claims 1 - 7 with a polymeric binder in an aqueous or solvent-based medium for application to metal foils and plates or for the production of coatings with an increased graphite/binder ratio and increased electrical and thermal conductivities, especially for the production of gas-tight and liquid-tight coatings on metal substrates.
11. Use of metal substrates coated according to Claim 10 as negative electrodes in lithium ion batteries.
12. Use of the graphite powders produced according to one of Claims 1 - 7 for the production of thermoplastic or thermosetting graphite/polymer composites which can optionally be further compressed to gas-tight graphite plates of high electrical conductivity, preferably for use as bipolar plates in polymer electrolyte fuel cell technology.
13. Use of the graphite powders produced according to one of Claims 1 - 7 for the production of metallic non-ferrous sintered materials.
14. The articles produced according to one of Claims 8 - 13.

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Figur 1