

The present invention relates to a sintered silicon carbide body, in particular for mechanical packing, and a packing comprising such a sintered body.

5 Silicon carbide-based materials have already been employed for sliding components: Japanese Patent JP-A-62 138377 proposes a composite consisting of a silicon carbide with high open porosity, into the pores of which a lubricant is introduced under pressure before a final drying operation. Furthermore, Patent Application
10 EP 0 361 326 proposes a structure consisting of silicon carbide impregnated with silicon containing carbon particles, out of which a sliding surface is produced and this surface is then given a surface porosity by oxidation at very high temperature to a depth lower than
15 500 μm corresponding to the pore dimension.

Mechanical packings are well-known fittings comprising two rings sliding one on the other to make a seal for the rotary shaft of a pump in automobile engines, for example.

20 Rings made of ceramic materials and, for example, of combinations of sintered silicon carbide for one ring and carbon-based material, in particular graphite, for the counter-ring, are increasingly being used for these packings.

25 A significant problem for this type of material is the wear on the sliding rings. In fact, if the wear is too pronounced, there is a risk of surface splintering being produced, in particular on the carbon-based counter-ring.

30 Irregularities or defects may also be seen to appear on this same surface, the flatness of which will thus be reduced.

The consequence of these surface defects is, of course, a reduction in the leaktightness of the system,
35 which gives rise to leakage of liquid.

The aim of the invention is, therefore, to develop a silicon carbide material which can be used in mechanical packings and is able to confer on the latter an improved resistance to wear, very particularly at high

speeds of rotation.

To this end, the invention proposes a sintered body based on silicon carbide which is free from impregnated silicon, as indicated in Claim 1.

5 After long-term tests (1000 hours) and at high speeds of rotation it has been possible to demonstrate a very good leaktightness of the packings using the sintered bodies of the invention.

10 Other characteristics and details of the invention will become more clearly apparent on reading the description which follows.

The sintered body of the invention is based on silicon carbide (SiC). It is possible to use α -SiC or β -SiC or combinations of these two polytypes in any proportions.

15 It is of course possible for the composition of the sintered body to contain, in addition to SiC, any of the customary and well-known sintering additives, such as, for example, carbon, boron and aluminium.

20 It should also be mentioned that the invention also applies to sintered bodies of the composite type, that is to say comprising SiC and, in addition, free carbon, in particular in the graphite form.

25 Thus, it is possible to consider composites comprising up to 48 % by weight of free carbon, in particular up to 20 % and more particularly up to 10 %, it being possible for this carbon to be in the form of a mixture of amorphous carbon/graphite carbon in any proportions.

30 These bodies of the composite type based on graphite are particularly valuable because they possess improved lubricant properties as a result of the reduction in the coefficient of friction.

35 Finally, the sintered body is based on a SiC which has a particle size of generally between 1 and 500 μm and preferably between 1 and 20 μm .

One of the main characteristics of the sintered SiC body according to the invention is that it is a porous body having a very specific average pore diameter.

This average diameter is that obtained on the basis of values measured by examination with an electron scanning microscope.

5 According to the invention this diameter must be between 60 and 200 μm .

It has been noticed that with an average pore diameter of less than 60 μm high wear was observed, at least on the counter-ring of the mechanical packing.

10 Moreover, with an average diameter of more than 200 μm the pores become too large and the sintered body has a reduced mechanical strength.

In general it is preferable to have a maximum value of the pore diameter of 150 μm , and thus to be within the range 60 - 150 μm .

15 It is for this reason that, according to various variants of the invention, sintered bodies having an average pore diameter of between 60 and 200 μm , and more particularly 60 and 150 μm and still more particularly 60 and 100 μm could be considered.

20 The pores of the body according to the invention are of a spherical shape or of a shape close to a sphere. This shape permits high porosities to be achieved, while ensuring complete leaktightness of the body.

25 Furthermore, these pores are essentially closed pores.

The porosity of the sintered body according to the invention is between 4 and 18 %.

30 This is understood to be the total porosity, that is to say that taking account of the open and closed pores. This porosity (P) is given by the equation

$$P = 100 - d \left[\frac{x}{2.25} + \frac{100 - x}{3.21} \right]$$

35 d being the apparent density of the body and x being the mass percentage of free carbon contained in the material; 2.25 being the density of the free carbon and 3.21 that of the silicon carbide.

If the porosity is less than 4 %, the resulting SiC body does not improve the wear resistance of the

mechanical packing. Above 18 %, leaktightness is no longer ensured and the mechanical strength is also too low.

5 Preferably, this porosity is between 5 and 15 % and more particularly 8 and 15 %.

 The sintered body of the invention may be obtained by a process which consists in adding a pore-forming material in the form of microbeads or granules to the mixture of products intended for the preparation of
10 the sintered silicon carbide.

 The pore-forming material may be used in several ways. For example, a slip based on SiC, sintering additives and a binder in water or in an organic solvent may be formed in a ball mill. The pore-forming material
15 is then added to this slip, with stirring. Finally, the product is dried by spraying.

 Another process consists in forming and then drying a slip of the type described above, but without the pore-forming material. The pore-forming material is then mixed dry with the dry product thus obtained.
20

 The pore-forming materials which can be used are well known. They are in general organic materials of the polymer type. Products obtained from an emulsion polymerization are preferably used. These materials must
25 not be soluble in water or the organic solvent used in the composition of the slip. They must also be able thermally to withstand drying, in particular by spraying.

 Examples which may be mentioned are polystyrene, polymethyl methacrylate and polyamide.

30 The dried product obtained is finally shaped, for example by isostatic or uniaxial pressing, to give a green article. Shaping may also be carried out by casting or extrusion.

 This green article is sintered in a manner known
35 per se.

 In general, the sintering temperature is between 1900 and 2500°C and more particularly between 2000 and 2200°C. Sintering is carried out under vacuum and/or under an atmosphere of a gas of the argon, carbon

monoxide or carbon dioxide, helium, hydrogen or nitrogen type, for example.

5 The invention also relates to the mechanical packings comprising a stationary ring and a rotary ring, at least one of which consists of the sintered body of the type which has just been described above.

10 The other ring may be produced from a carbon-based material and in particular resin-impregnated carbon. This material may also consist of silicon carbide or silicon nitride; it may also be a sintered alumina body. It may also be a material of metallic type, for example carbon steel.

15 Finally, it is possible to use a packing in which the two rings would consist of the sintered body according to the invention.

 The sintered body of the invention may also be used in bearings.

 Of course, the sintered body of the invention may be used in applications other than mechanical packings.

20 Thus, it may be used in any friction system of, for example, the thrust block, bearing or slide type comprising at least one friction part consisting of the said body or part of which consists of the said body. It may thus be used, for example, in bearings and in the following configurations: SiC bore - metal shaft, SiC bore - SiC shaft, SiC bore - metal shaft with SiC or graphite liner.

25 An example will now be given.

EXAMPLE 1

Preparation of the rings

Two slips are formed containing 60 % solids in water comprising inorganic matter, a phenolic resin providing carbon after pyrolysis and 3 % of polyethylene glycol.

The proportions of the constituents of the slip are the following, in parts by weight:

		<u>Slip 1</u>	<u>Slip 2</u>
10	SiC	99	99
	B	1	1
	Pore-forming agent:		
	Plexigum M 914	6	-
	Orgasol 2002 ES 3	-	1.6
15	Carbon		
	("ex phenolic resin")	1.5	1.5

Plexigum M 914 is a polymethyl methacrylate with an average particle size of 90 μm . Orgasol 2002 ES 3 is a polyamide with an average particle size of 30 μm .

The silicon carbon [sic] has a specific surface of 12 m^2/g and an average diameter of 1.3 μm .

The pore-forming agents have been mixed with the slip with stirring.

The slips are then dried by spraying.

The powders obtained are then pressed at 1500 bars to give green parts. After a heat treatment under nitrogen at up to 600°C, which in particular permits the removal of the pore-forming agent, the said green parts are then sintered at 2070°C under argon for 1 hour.

A ring 1 (resulting from the first slip) according to the invention and a ring 2 (resulting from the second slip) which serves as comparison are obtained. The characteristics of the products are given below:

Ring	1	2
Total porosity %	14.3	5.3
Average pore diameter (μm)	80	25
Apparent density g/cm^3	2.75	3.04

Tests

Rings 1 and 2 are tested as a rotary sliding ring in a mechanical packing. The fixed sliding counter-ring is made of graphite carbon.

The test conditions are as follows:

- temperature : 130°C
- fluid : pure antifreeze
- speed of rotation : 6700 rev/min
- pressure : 1 bar
- test period : 1000 hours.

In the case of the packing fitted with ring 2, pronounced undulations are observed on the surface of the counter-ring after the test. These undulations have a height which can reach 0.8 to 1.2 μm .

In contrast, in the case of the ring 1 according to the invention, the undulations on the counter-ring are very small, that is to say they have a height of at most 0.1 μm .

After a static leaktightness test at 20 bars, ring 1 is leaktight, which shows that the porosity is entirely closed.

EXAMPLE 2

A slip with a solids content of 60 % is formed in water comprising inorganic matter, a phenolic resin providing carbon after pyrolysis, 3 % of polyethylene

glycol and a Plexigum M 820 pore-forming agent.

The proportions of the constituents of the slip are the following, in parts by weight:

5	SiC	:	96.37
	B ₄ C	:	0.63
	Graphite carbon	:	3.00
	Pore-forming agent	:	4.1
	Carbon "ex phenolic resin"	:	1.5

10 "Plexigum M820" is a polymethyl methacrylate whose average particle size is 75 μm .

The graphite employed is the Lonza grade KS6, whose specific surface is 20m²/g and average diameter approximately 3.5 μm .

The silicon carbide is the same as in Example 1.

15 The pore-forming agent is mixed with the slip, with stirring.

The slip is then spray-dried.

20 The granules obtained are then pressed at 1500 bars to give a green article. After heat treatment under nitrogen at 600 °C, which permits the removal of the pore-forming agent and the conversion of the phenolic resin to carbon, the article is next sintered at 2070 °C under argon for 1 hour.

25 An αSiC /graphite composite material is obtained, whose characteristics are given below:

	Total porosity (%)	:	10.7
	Average pore diameter (μm)	:	65
	Apparent density (g cm ⁻³)	:	2.83
30	Theoretical density excluding porosities (g cm ⁻³)	:	3.17



CLAIMS

1. Sintered body based on silicon carbide which is free from impregnated silicon, characterized in that it has pores which have an average diameter of between 60 and 200 μm and a total porosity of between 4 and 18 %.
2. Sintered body according to Claim 1, characterized in that the average pore diameter is between 60 and 150 μm .
3. Sintered body according to Claim 2, characterized in that the average pore diameter is between 60 and 100 μm .
4. Sintered body according to one of the preceding claims, characterized in that it has a porosity of between 5 and 15 %.
5. Sintered body according to Claim 4, characterized in that it has a porosity of between 8 and 15 %.
6. Sintered body based on silicon carbide according to one of the preceding claims, characterized in that it additionally comprises free carbon, it being possible for this free carbon to be in graphite form.
7. Mechanical packing, comprising a stationary ring and a rotary ring, characterized in that at least one of the rings consists of the sintered body according to one of Claims 1 to 6.
8. Device of the thrust block, bearing or slide type, comprising at least one friction part consisting of a body according to one of Claims 1 to 6 or part of which consists of the said body.