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SELUSIC

PROJECT COORDINATOR: F. Burgmann / FB

PARTNERS:
Ceramiques et Composites / C&C
Hydromecanique et Frottement / I+EF.
Lowara
Fraunhofer-Institut fur Lasertechnik / ILT

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DEVELOPMENT OF SELF-LUBRICATING SINTERED SILICON CARBIDE WITH OPTIMIZED POROSITY AND INFILTRATION

SELUSIC

Authors (Name / Company)

R. Schicktanz
Feodor Burgmann, D-82502 Wolfratshausen

M. Bougoin
Ceramiques & Composites, F-65460 Bazet

Ph. Maurin-Perrier
Hydromecanique et Frottement, F-42166 Andrézieux-Boutheon Cede.

A. Janigro
Lowara, I-36075 Montecchio Maggiore

K. Wissenbach, W. Barkhausen
FhG-Institut fur Lasertechnik, D-52074 Aachen

ABSTRACT

It is widely recognized that ceramics are not able to run in dry friction without surface damage. It was shown that at a high level of porosity of the ceramic material, the pores in the sliding surface of mechanical seals or sliding bearings store the lubricating medium or a special lubricant, thereby enabling increased periods of dry running.

The influence of the pore shape, pore size, pore distribution and amount of porosity on the tribologic behaviour is clarified in this work. The manufacturing process of the SSiC showing this controlled porosity was optimized and a set of impregnants to be processed in this porous material was developed. Performance in mechanical seals, water pumps and sliding bearings for magnetic couplings is assessed. Laser beam treatment was applied for the optimization of the surface structures on SSiC ceramics.

Objectives

The main objectives of this project are to develop a self-lubricating SSiC material to improve the reliability of SSiC components when subjected to dry friction and to extend the spectrum of ceramic components such as sliding bearings and mechanical seal faces.

SSiC in the form in which it is currently available is just able to withstand a dwell time under dry friction conditions of less than 10 minutes. Modified materials will be able to attain dry friction periods of several hours.

Role of the partners in the course of the project.

In this project five partners (see first page) cooperated highly interdisciplinarily in order to achieve the objectives. The partnership structure included:

- one ceramic manufacturer (C&C), responsible for defining the requirement profile of SSiC-structure, manufacturing and finishing of test samples, development of a new manufacturing process of the improved material and prototype manufacturing

-one research and development centre for laser technology (ILT), responsible for structuring of SSiC surfaces by laser radiation.

-one research centre, specialized in the study of phenomena that lead to surface damage of mechanical parts by friction (FIEF), responsible for surface investigations and the development of self-lubricating SSiC-material.

-one manufacturer of mechanical seals and sliding bearings (Burgmann), responsible for defining the requirement profile of SSiC-structure, screening, dry-running and bearing tests, definition of test samples and testing of the improved material in seals and bearings.

-one end user manufacturing pumps (Lowara), responsible for testing components for reference purposes, application-oriented tests and field tests.

This means that the project covered all development phases of the SSiC material, from the manufacturing process to industrial application, analysis of the surfaces, development of the infiltration process and optimization of the SSiC structure by means of laser treatment. The results of this collaborative work will be presented by following this summarized sequence.

Results

A. Preliminary requirement profile for surface structure of SSiC materials

On the basis of literature and patent research work carried out the wear characteristics of various SSiC qualities available on the market were correlated with the surface quality of the sliding surfaces. This resulted in a preliminary range of requirements regarding pore size, pore shape and pore distribution, which served as the starting point for laser-beam structuring operations.
The requirement profile relating to the surface was given as follows:

- Angular shapes must be avoided.
- The pore shape should be nearly a half-ellipsoid, possibly a half sphere.
- One of the main objectives for the following work was to find out a relationship between the apparent pore diameter (Φ) and the friction behaviour; consequently it was necessary to start with a unique pore size in each type of material.
- Several figures had to be tested with an apparent pore diameter (Φ) ranging between 10 and 200 pm.
- The amount of porosity is defined as the ratio between the "porous surface" $[\Sigma(\pi.\Phi^2/4)]$ and the total related surface.
- Several figures had to be tested with the amount of porosity ranging between 2.5 and 200A.
- For a given pore size the amount of porosity can be varied by varying the number of pores per unit area.
- The distribution of the pores in the surface had to be as close as possible to a random one. In particular, the pores must be out of alignment with the friction direction.

B. Manufacturing, finishing and analysis of test samples

Two different materials were produced : the C&C SiC-100 and the C&C SiC-150. The first one is the standard non-porous sintered silicon carbide of C&C while the second is the standard grade showing a controlled porosity.

[n order to optimize the SSiC structure, test samples were produced under controlled conditions. The preparation of these samples included the following steps :

- preparation of ready to press (spray-dried) granules including forming and sintering additives,
- forming : uniaxial or isostatic pressing + green machining,
- debinding and sintering,
- surface processing: grinding or lapping + polishing.

The surface topography of the samples was analysed.

C. optimization of SSiC surface structure by laser treatment

As a consequence of the exact controllability of the processing parameters, pores of varying size and shape can be produced by laser treatment. Therefore the optimization of the SS iC-structure will be carried out with laser treatment.
Structuring of SSiC surfaces is carried out with an excimer laser with a wavelength of l = 248 nm. The experimental set up is shown in Fig. 1. A mask with holes of defined diameter and distribution is positioned in the beam path. The pore depths depend mainly on the number of laser pulses, irradiated to the surface N and on the fluence F. The pore diameters are given by the diameter of the holes in the mask D_{mask}, the focal length of the imaging lens f and the reduction scale m. When varying these processing parameters F = 10-20 J/cm^{z}, N = 75-900, D_{mask} = 0.4 - 2 mm, f = 50, 100 mm and m = 1:10 or 1:20 different pore geometries listed in Tab. 1 are generated. Typical removal rates in the range of 0.1 up to 0.25 µm/pulse and accuracies of the pore geometry (diameter / depth) within some µm have been realized with this set up.

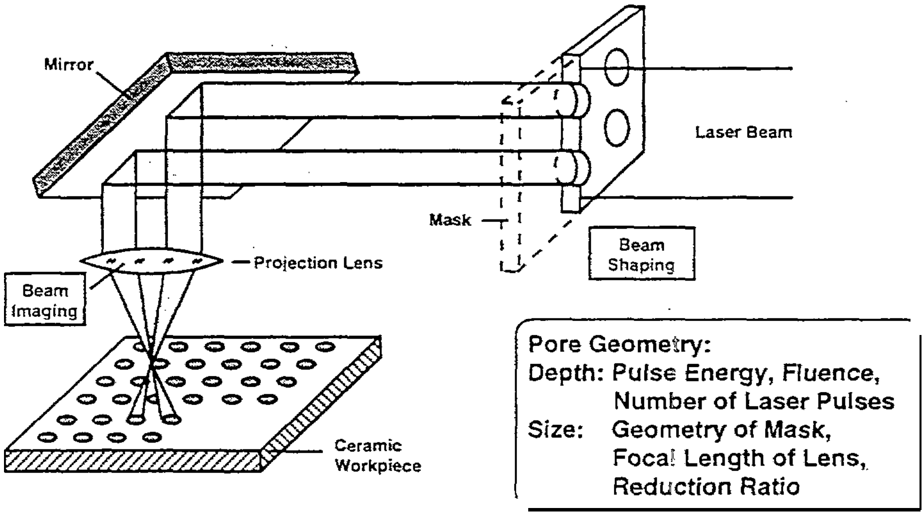

Fig. 1: Experimental setup

Pore diameter d [µm]	Pore depth t [µm]	Pore content p [%]
20	10	2.5/11.25/20
50	25	2 / 5 / 8 /12/16/20
50	25	2.5/11.25/20
110	55	2.5/11.25/20
200	100	2.5/11.25/20

Tab. 1: List of generated pore geometries

Surface characterization.

The pore shape is influenced by the fluence distribution on the surface. The pore shape is nearly cylindrical for pore diameters > 50 µm (Fig. 2) and hemispherical for diameters in the range of 20 pm.

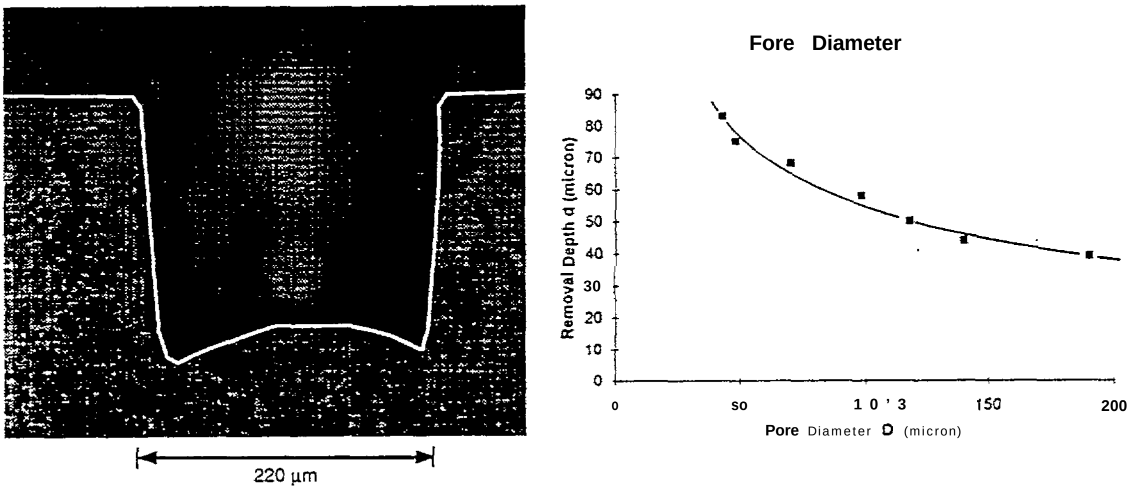

Fig. 2: Cross section of a laser generated pore/
Removal depth as a function of pore diameter

Pore diameter and degree of porosity were determined with three different measurement methods:

o Optical microscopy (ILT)
o 3-D surface analysis (HEF)
o VIDAS, video analyzing system (ILT).

Machining of seal rings.

For the production of seal rings a workstation including a KrF - Laser (Lambda Physics, EMG 201 MSC) was set up, which allowed seal face structuring with flexible pore geometry and distribution. An on-line energy measurement ensured constant laser pulse Energy E on the surface.
Machining of seal faces was carried out with the mask projection system (Fig. 1). Pore diameter and distribution are controlled by the geometry of the imaging mask. Different masks for variable diameters and porosities were cutted with high accuracy by a Nd:YAG laser. The pore depth was controlled by the number of laser pulses and the fluence.
The number of pores per seal ring depends on the pore diameter and degree of porosity. The required numbers of pores Npore on the complete sliding face for different porosities are between 400 (200 pm, 2.5 %) and 320000 (20 pm, 20 “A).

In order to reduce processing time a certain number of pores Nsim - depending on pore diameter and degree of porosity - can be processed simultaneously. In one processing step on a seal area of 600 µm x 1500 µm (m = 1:1 O) or 250 µm x 500 µm (m = 1 :20) simultaneous pore machining can be realized. Nsim are between 2 (200 µm, 2.5 %) and 118 (20 pm, 20 %).

The surface of the seal rings showed redeposited material in the vicinity of the pore. This redeposition was removed by a postprocess cleaning procedure by alcohol brushing, high pressure water stream and ultrasonic cleaning in alcohol.

Screening tests (FB):

The laser treated seal faces were installed in a standard seal (Fig. 3) and tested in demineralized water. A sudden increase in the power consumption of the test rig indicated increased friction due to dry-running. The operating limits of the tested sealing rings were reached. The test run was stopped immediately to avoid the complete destruction of the sealing faces.

Screening Test Mode

- **30 min run at 5 bar, linear temperature increase from RT to 60°C**
- **1 h linear pressure increase from 5 to 90 bar, temperature constant at 60 °C**
- **1 h linear temperature increase from 60°C to 90°C, pressure constant at 90 bar**

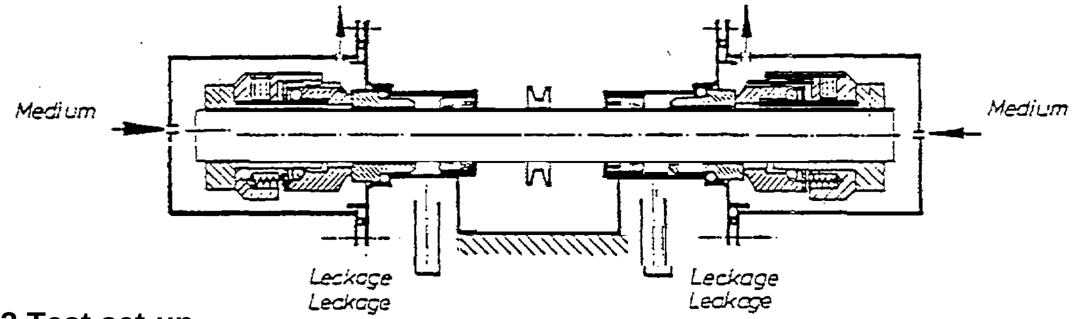

Fig. 3 Test set-up

For the final rating and decision about the process parameters for manufacturing SSiC with a calibrated pore size and a defined porosity the following correlations, restrictions and demands were taken into account:

- **Pore size ⇒ material strength ↓, heat conductivity ↓**
- **Porosity ↑ ⇒ material strength ↓, heat conductivity ↓**
- **Pressure ↑ ⇒ Power consumption ↑**
- **Leakage as small as possible**
- **smoothening effect of the seal faces and measured seal face temperature as small as possible**
- **Small sealing widths (≥ 2 mm) are restricting pore size and porosity**
- **Power consumption (friction coefficient, temperature in the sealing gap)as small as possible**

After analyzing and rating the results of the screening tests and having in mind the above mentioned topics the pore size range and the porosity were defined.

A theoretical analysis was carried out with the help of the Technion in Haifa, confirming the selected material porosity.

D. Development of self-lubricating SSiC materials

The objectives for HEF in the project were to develop a set of impregnants to be processed in a SiC material with optimized porosity, and to assess the performances of the association SiC/impregnant in model contacts (sphere on disc and small face seals) under various external solicitations (speed, type of lubrication, temperature of fluid, time). The impregnant is designed to remain solid when the fluid is present in the sliding contact and turn to a liquid state when the temperature is increasing in relation with an increase of friction coefficient.

Development of the impregnation:

The impregnation was processed with 3 different routes during the project:
immersion in melt product
immersion in a mixture of solvent and product
mechanical impregnation with a mixture of glass balls and product.

Due to the small. quantities of pieces to be treated during the project the first route was found to be the most efficient and time saving. For future industrial exploitation the last route is likely to be considered. During the project 59 different impregnants were developed.

Tribological tests on face seal contact:

Tests were carried out on the most interesting impregnants with a configuration disc on disc which was close to actual sliding contacts. This test enabled to run in fluid lubrication, dry friction or any intermediate case (with cycles of running alternatively in water and without water with different cycle times and total duration). This feature allows to simulate the case of a pump when no more pumping fluid is present at the inlet of the pump.

Test with non impregnated SSiC:

It is of major interest to know what is the lifetime of a mechanical seal starting from the moment when there is no more lubricant present in the contact. The test was carried out under fully floaded conditions with the contact immersed in water for 15 minutes. Then the vessel was drained out. The test was ‘stopped when a sharp increase of friction coefficient µ was detected. This increase of µ corresponds to the moment when there is no more water in the contact. These tests clearly demonstrated the advantage of using a porous SSiC material with a lifetime after start of dry friction of almost 11 minutes compared to only 2 minutes with classical dense SSiC material.

Tests with SSiC with superficial closed porosity:

Using this procedure tests were started with the contact fully immersed in water at 80°C.. Water was kept for 25 minutes, then the vessel was drained out and the system was run in dry conditions for 5 minutes. The above described cycle was repeated up to 7.5 hours (or lubricant failure}.

The following conclusions can be drawn :

no impregnant provides a lifetime longer than 4 cycles

the experiment carried out in cold water (20°C) showed clearly that the short lifetimes observed are related to the fact that most of the impregnant is washed out from the pores during the stage of running where hot water is in the contact. This fact is related to the value of the drop point of the impregnants which is in the range of 50 °C to 100 °C for the impregnants that are efficient in dry friction.

The conclusion of this series of tests is that an impregnant with a low drop point is required to operate under dry friction. The problem experienced is that in such case the volume of impregnant on the surface porosity is not sufficient to work for more than 4 cycles wet/dry in hot water. One has to consider that porous SSiC material have to be used with impregnation either in pure dry friction (applications could be face seals for compressors where a gas has to be sealed), or in mixed wet/dry friction with water at ambient temperature (below 40 'C).

Tests with SSiC with open porosity:

A new material was developed by C & C. This material achieves an open porosity. The interest of such a porosity is to increase the volume available either for water when the material is run without impregnation, or for the impregnant. Tests evidenced that for dry/wet cycled tests there is no time limitation for this new material with or without impregnation. This is likely to be related in the case of no impregnation to the fact that water is retained inside open porosity during the wet stage and is slightly released during the dry stage. In the case of impregnation the increased impregnated volume is certainly responsible for the increase of performance. Continuous dry tests demonstrated that friction coefficient is stable at an average value of 0.13 for at least 100 hours.

The following conclusions could be drawn :

SiC material which exhibits a level of porosity above the interconnecting threshold has demonstrated an increased performance compared to a SiC material with closed porosity.

Such a material could be considered for applications where no tightness is required and where mechanical solicitations' are compatible with its slightly reduced mechanical properties.

When no leakage through the component is required one can imagine to perform a first impregnation to a low volume with a thermosetting resin and then to, carry out a second stage with the lubricant acting impregnant.

Dry-running wear tests:

Dry-running **wear tests** (up to 350°C) **have been sub-contracted to LMCTS :** Laboratoire **de** Matériaux Céramiques et Traitements **de Surface,** ENSCI Limoges **France, Dr F. PLATON.**
A first range of tests were performed in the following conditions :

- **disk on disk: 28 mm id., 36 mm o.d, 32 mm average diameter**
- **axial load = 135.6 N i.e. axial pressure = 0.34 MPa**
- **speed = 370 rpm # 0.620 m/s**
- **temperature: 20, 200, 300 and 350°C**
- **duration : up to 5 hours**
- **sliding material :** C&C **SiC-150 lubricated with 6 products selected among** HEF **developmental lubricants :15, 121, 122, 123, 151 and 152; each material is tested against itself.**

The RESULTS of these low speed tests are summarised in the following table which shows the superiority of the 122 lubricant.

. Lower friction coefficient of 15 and 121 at 20°C
. Good behaviour of all lubricants except 151 at 200°C, 5 hours
. lower friction coefficient of 15 and 122.
• Good behaviour **of 122 at 300°C, 5 hours**
. Poor behaviour **of 122 at 350°C, after 45 mn**

[n a second range of similar dry-running tests the three waxes 121, 122 and 123 showed a good lubricating effect at 5 m/s during 1 hour but **a poor one when the speed was increased to 10 m/s.**

The same HEF developmental lubricants (15, 121, 122, [23, 151 and 152) were investigated by TGA and DTA **thermal analysis. These measurements gave evidence of their melting point. A beginning of thermal decomposition was also detected, always above** 300°C.

The melting point was also determined by the measurement of the viscosity versus the temperature.

The nettability angles of a drop of liquid **wax** on **a SiC-150 ceramic (at** 20°C) **were measured for the lubricants** 15, **121, 122 and 123. All these waxes showed a good nettability and no significant difference : all angles were comprised between 14 and** 17°.

In **spite of its low melting** point **(around** 60°C), **the** 122 wax **gave good and best results in these relatively short duration dry-running tests:**
. low friction coefficient at up to 300°C, **low speed,**
. good friction behaviour **at up to** 5m/s, 20°C.

Burgmann **tested the Impregnation 122 at** start/stop cycles **and temperatures up to** 300°C **in the high speed application of a dry-running gas** seal.
The test conditions are shown in the following table:

Test rig		High-speed test rig HG5
Test seal		DGS5/123-Ta1
Material combination		SiC 150 with I22-Impr. / SiSiC
Speed-	(rpm)	0-15000
Sliding velocity	(m/s)	0-112
Medium		Pressurized air
Temperature of gas in the sealing gap	(°C)	60-300
Temperature of gas in seal chamber		60-196
Pressure	(bar)	o-75
Running time/pressure step		8h at 75 bar / 6h at 0 bar
Start- Stop cycles/pressure		50x at 0,40,20.30, 50 bar

Table 3 Test conditions of the Dry-running Gas seal test

Result:

- The impregnation I22 is suitable for this application together with gas temperatures of about 200°.

Dry running tests in sliding bearings:

Dry running tests with the Lubricants [21 and I51 were carried out in the sliding bearing of a magnetic coupling. For testing the basic lubrication properties of these two selected impregnants, C&C sampled Burgmann with SiC1 00 bearing parts in which several holes were drilled in the green stage of SSiC-production, thus providing a large amount of impregnant in the sliding area.

Result:

Impregnant:	Running time before failure:	Remark:
I21	0,45 h	Axial load up to 1175 N possible , Impregnant provides very good lubrification
I51	·0,21 h	No axial load possible, Impregnant provides poor lubrification, stays in the holes.

With this increased amount of I21 lubricant good results under pure dry running conditions were achieved. With customary SSiC-bearings an axial loading of the sliding bearing is not possible up to this degree under dry running conditions. The lubricant I51 showed no or very little lubrication properties under these test conditions.

E. Development of a new manufacturing process

The main objectives were to obtain . . .

- a narrow distribution of the pore size
- if possible, several grades with an average pore diameter ranging from 20 to 200 µm
- nearly spherical pores

- several grades with an additional porosity (from polymeric spherical particles) comprised between 2.5 and 15 % (if possible)

Around 20 different commercial powders were evaluated as a fugitive pore-forming polymer. These polymers were mainly investigated by...

- scanning electron microscopy (S. E. M.)
- laser grain size analysis
- thermogravimetric analysis (TGA)
- laboratory mixing test with SiC slurries
- laboratory die-pressing test of the SiC-polymer mixture

Two grades « L2 » and « P1 » were selected as the most promising. Furthermore, « L2 » and « P1 » appeared complementary as they covered different grain size areas. Unfortunately, their grain size distribution was too large and it was thus necessary to make a grain size selection. The following 4 grain size ranges were obtained :

- with L2, named « L2/50 » because its median diameter was nearly 50 µm,
- with L2, named « L2/70 » : median diameter nearly 70 pm,
- with PI, named « P1/100 » : median diameter nearly 100 pm,
- with P1 named « P1/140 » : median diameter nearly 140 pm.

At this laboratory stage, several batches of ready-to-press powders were prepared by introducing one of the selected fugitive polymer. Several levels of polymer content were investigated in order to obtain a porosity (from polymer) of 3.0, 5.0, 7.5 or 12 %.
Tiles were shaped either by iso-pressing and by die-pressing the above ready-to-press powders and sintered. Standard test-pieces (50x4x 3 mm^3) were then sliced and machined from these porous SiC parts for the measurement of their 4-points bending strength.

The total porosity of the sintered ceramics was typically 3 % more than the level which was expected from the fugitive polymer content. This is due to the residual closed porosity of the silicon carbide.

The 4-points-bending strength of the porous SiC was around 50% that of the non-porous one. It slightly decreases when the pore diameter is increased; but it is strongly lowered by high porosity levels. Typically, this strength can be kept over 200 Mpa if the average pore diameter is lower than 100 µm and the porosity of the polymer is not in excess of 5%.

The Weibull modulus is increased from 12 for the non-porous SiC to' over 20 with an addition of the « L2/70 » polymer, up to 7.5 vol. %.

Manufacturing, finishing and analysis of prototype ceramic parts for tests : Definition and manufacturing of test samples:

Over 130 parts specified by BURGMANN were manufactured, starting from the pilot batch of powder produced here above. They were delivered with « Ref. 155 » as the material reference .The industrial feasibility of complex shapes was demonstrated with this new ceramic. The characterisation of the « Ref. 155 » SiC (see Fig 4), the new control led porosity SSiC was completely achieved. Two kinds of measurements were subcontracted in French Universities. The thermal conductivity was measured with a flash-laser method, from 20 to 1300°C. The elastic modules and Poisson's ratio were obtained from an ultrasonic method.

Compared with the C&C SiC-100 (non-porous sintered silicon carbide), this « Ref. 155 » SSiC shows only slight differences.
. a 4% lower density,
. a 14% lower thermal conductivity,
• a less than 10% lower Young's modulus.
The major difference is related to its mechanical strength which is weakened by around 40 % but this loss is compensated by its doubled Weibull modulus.

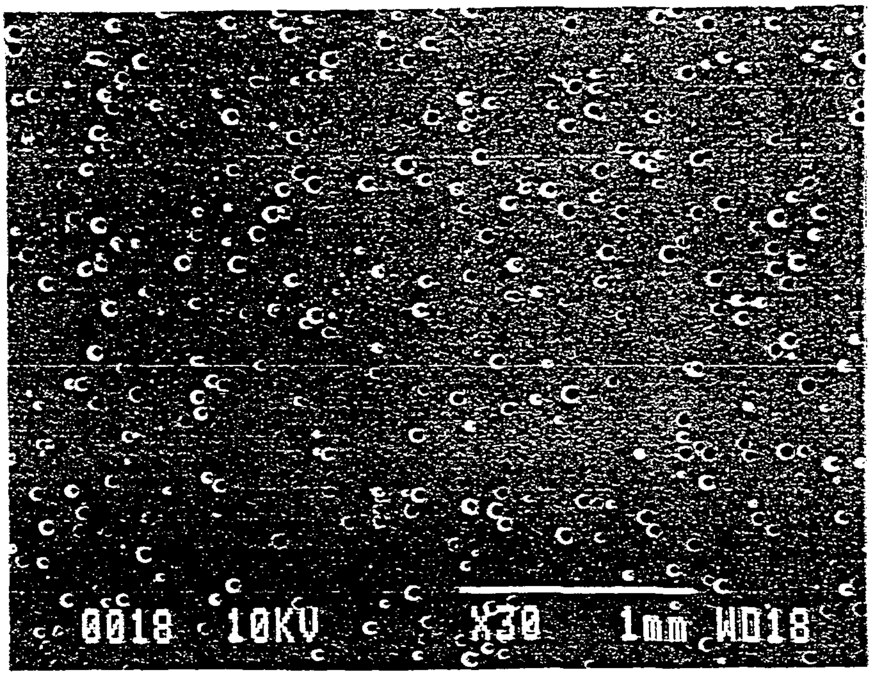

Fig. 4 S.E. M view of a typical SiC155 polished surface

F. Bearing test of the optimized materials

The new material SiC1 55 was tested in the magnetic coupling test rig. The results were compared with the already available test results.

Dry running tests:

. The bearing parts behave similiar to the results with the drilled bearing parts. No real difference could be detected.
. No difference between the impregnants I21 and 122 could be detected.

SiC 155 without impregnation could be operated up to 20 minutes before torque oscillations occured, which means that it is superiour to non porous material, which fails after about 10 minutes of operation.

Lubricated bearing tests:

Tests with the same materials as tested under dry friction were carried out under lubricated conditions.
. The material SiCl 55 shows an excellent running behaviour.

- **With this material the maximum radial forces can be increased by at least 20%, which rises the operating limits considerably.**
- **At high temperatures the decrease of the maximum loads, which are caused by partial dry running conditions in the contact area, is less compared to the dense SiC100 material. A similiar effect can be recognized with SiC155.**
- **Compared to SiC 150, SiC155 shows much higher operating limits.**

E. Application-oriented tests and field tests

The operating behaviour of seal faces and bearing components consisting of SSiC with a defined and impregnated porosity were checked on test rigs and water pumps. The results of these tests will be compared with the results of the performance analysis. In addition to the standard material tests, long-time tests at 1 MPa were carried out.

Test rig		**High-pressure test rig**
Test seal		H74N/53
Material combination		SSiC/Carbon grafite; SSiC/SSiC
Speed	(rpm)	3000
Sliding velocity	(m/s)	9,5
Medium		**Demineralized water,** Oil
Temperature	(°C)	60
Pressure	(MPa)	1.0; 2.5; 5.0; 7.5; 10.0; 13.0
Running time/pressure step	**(h)**	50
Total running time	**(h)**	300 /1000

Table 4 Test parameters

Results:

Up to medium pressure ranges of about 7.5 Mpa the improved material SiC155 showed the best results concerning the wear rate of the mating carbon grafite ring.

The seal face combination SiC155/SSiC proved to be the best sealface combination for the application in oil as lubricant in terms of wear of the seal faces and friction losses.

Test results in pumps at Lowara test facility:

The conditions of service encountered in water pumps for the residential and light industrial market segments are considered not particularly severe for the mechanical seal. Therefore, the installation of mechanical seals with SiC/SiC faces is normally considered an added cost without additional benefits for the pump user.

However, the duty may become substantially more severe for the seal in case of intermittent operation of the pump, with poor or marginal seal flushing. Eventually the seal may run dry, which is clearly an abnormal, but possible, operating condition.

Test results performed with enhanced silicon. carbide seal faces (solid/solid & solid/porous) indicate that both combinations, using SiC 100 as base material, have an improved capability of dry running with respect to commercially available grades.

Owing to its lower coefficient of friction the solid/porous combination is less subject to the adhesion phenomenon (a frequent occurrence in the field that is considered the major drawback of SiC/SiC faces) and it is therefore more attractive for water pumps.

In conclusion, the enhanced SiC material resulting from the SELUSIC Project showed very positive behaviour when applied to water pumps of small to medium size, both in terms of extended dry running capabilities and reduced adhesion. It is foreseeable that the use of the solid/porous seal face combination will be generalized for water pumps in the next three to five years in the water pump market.

5- CONCLUS1ONS

The main objectives of this project were to develop a self-lubricating SSiC material to improve the reliability of SSiC components when subjected to dry friction and to extend the spectrum of ceramic components, such as sliding bearings and mechanical seal faces.

It was demonstrated that SiC sliding faces can be structured in a defined and reproducible way by means of excimer laser beam treatment. Such surface structured SiC sliding faces improve the operating behaviour of the ceramic parts.

An impregnant with a low drop point is required to operate under dry friction. SSiC material with closed porosity show limited service life in hot water. These materials have to be used with impregnation either in pure dry friction or in mixed wet/dry friction with water at ambient temperature. Tests with a new material that achieves an open porosity showed that for dry/wet cycled tests there is no time limitation due to the increased volume of impregnant.

A sintered silicon carbide showing a controlled and spherical porosity was developed. The manufacturing process of the controlled porosity SSiC was optimised and the industrial feasibility of complex and possibly large components with this new material was demonstrated. This new SiC exhibits physical characteristics similar to that of the non-porous SSiC. It shows a reduced mechanical strength, but this loss is compensated by a doubled Weibull modulus which proves the higher reliability of the new ceramic.

Tests in sliding bearings and mechanical seals evidenced that this new SiC material has improved running characteristics and extends the operating limits of sliding bearings and mechanical seals in dry friction and in low and high viscuous media. This material can be used in random lubrication without impregnation.