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## ABSTRACT

Many of the materials which have been developed for use as particle filters in the exhaust of diesel engines have characteristics which give rise to significant problems in practical use. Due to its special characteristics, it is shown that SiC is very well suited for use as the base material for particulate filters. The physical and thermal properties of porous SiC substrate material as applied to diesel particulate filters have been determined and are presented. Experimental results from several types of filter regeneration processes in exhaust gas systems confirm the improvements in the area of thermal load and reduction in temperature level during regeneration. The reduction in temperature during regeneration is shown to be consistent with the high thermal conductivity of SiC.

## INTRODUCTION

In recent years, concern over the adverse health effects of particulate matter in diesel engine exhaust gasses has led to increasingly stringent standards for exhaust emissions. In addition to engine modifications and improved engine control, aftertreatment with particulate filters has been investigated for the reduction of particulate emissions from diesel engines.

There is a variety of filter types that have been proposed and tested on engines and in vehicles, basically differing in the type of material used and the physical structure of the filter. Often these two variables are interrelated.

One of the most common types of filters to date has been the so-called "wall flow filter", which is normally constructed of a honeycomb structure made of a porous, sintered, ceramic material. Although most filters could be thought of as a kind of wall flow filter, this designation normally refers to the filter with the honeycomb structure with plugged alternate ends (1)*. The most common version of this type of filter is made of Cordierite, although recently Silicon Carbide (SiC) has also been used as a filter material (2,3). This type of filter has the advantage of a high filtration efficiency, which remains high as long as the total structural integrity of the filter in maintained. It is possible to coat the surface of the filter with materials that are catalytically active in order to reduce the ignition temperature of the accumulated soot (4).

Another type of particulate filter that has been investigated is the ceramic foam filter, in which the filtering element is a hollow tube made of a porous ceramic material, Mullite in Reference (5) and SiC in Reference (6). This type of construction suffers from a relatively low surface to volume ratio, which leads to higher filter volume and the need for more frequent regeneration.

A somewhat similar concept is the use of stainless steel tubes with a series of holes in them, which are wound with fine glass fibers to make a filtration element (7). This type of filter can have a high filtration efficiency, but since the filtration structure itself is not bonded, it is susceptible to long term mechanical degradation due to vibrations and alternating thermal loading. This type of filter is often used with some kind of fuel additive or ignition promoter that is periodically sprayed onto the filter to reduce the regeneration temperature (8).

The use of a stainless steel wire mesh has also been investigated as a filter material (9). This type of filter has only been used in connection with catalytically active coatings to combine the filtration process with catalysis in the so-called "Catalytic trap oxidizer". One of the primary disadvantages of the systems has been a low filtration efficiency compared to other systems.

* Numbers in parentheses indicate Reference at end of paper.

Of all these systems, the one that has probably shown the greatest potential and amount of study is the wall flow filter based on the honeycomb structure. This is due mainly to its compact structure, high filtration efficiency, and general mechanical stability. There are some problems that have not been completely solved with this filter. These are mainly due to the type of material that has traditionally been used. Cordierite is a low cost ceramic material, but has some properties that are undesirable in a diesel particulate filter. The first of these is a low melting point, which can be exceeded in practical engine conditions if a filter regeneration occurs at an unintended time. This can result is a very rapid destruction of the filter. A large portion of the development effort for Cordierite wall flow filters has been to develop systems that are prevented from regenerating under such conditions.

An additional characteristic that contributes to this problem is the low thermal conductivity. This results in locally high filter temperatures because the heat transfer through the filter substrate is too low to reduce the temperature resulting from the combustion of the particulate matter. This is especially undesirable in connection with the relatively low melting temperature. Filter meltdown often occurs in low flow conditions, where there is not sufficient flow to remove the energy released by combustion through convection.

The low thermal conductivity is also a problem in electrical regeneration that is often used for fleet and off road vehicles. An example is the "City Filter" concept, where a filter is regenerated after typically a day's service by electrically heating the filter of a parked vehicle to a temperature adequate to ignite the particulate matter (10). The low thermal conductivity of the Cordierite makes it more difficult to heat the filters.

## SIC AS A FILTER MATERIAL

Silicon carbide is a material that has the potential to solve some of the problems that arise during the use of the wall flow filter. Table 1 presents a comparison of the properties of pure polycrystalline SiC, Cordierite, and Mullite. It can be seen that for the case of pure, solid materials, the SiC has a decomposition temperature of 2300°C, while the Cordierite melts at a temperature of 1350°C, and Mullite at 1850°C. In practice with actual filters, these respective temperatures are more nearly 1600, 1200 and 1400°C. This is one of the main advantages of SiC, since a material with such a high decomposition point should be able to withstand almost any kind of regeneration process, intended or unintended, without being destroyed. Such a characteristic should enable the use of a cheaper and less complicated control or security arrangement, since a pure thermal destruction of an SiC filter is highly unlikely.

| Property for Pure Materials | Silicon Carbide | Cordierite | Mullite |
|---|---|---|---|
| Formula | SiC | $2MgO2Al_2O_35SiO_2$ | $3Al_2O_32SiO_2$ |
| Density, $\rho$ | 3.21 | 2.1 | 2.9 |
| Flexural Strength, $\sigma$, MPa | > 380 | 195 | 200 |
| Young's Modulus, E, GPa | 410 | 132 | 152 |
| Poission's ratio, $\nu$ | 0.15 | 0.25 | 0.2 |
| Specific Heat, J/kg-°C | 670 | 1000 | 850 |
| Thermal Expansion, $\alpha_T$, 1/ °C 25-500 °C | $3.6 - 4.5 \times 10^{-6}$ | $0.3 - 1.0 \times 10^{-6}$ | $4.2 \times 10^{-6}$ |
| Thermal conductivity, $\lambda$, W/m-°C | 100 | 2.8 | 4.5 |
| Thermal Shock Parameter, $TSP_1$, $\frac{MOR}{E\,\alpha_T(\Delta T)}$ | 0.25 | 2.5 | 0.3 |
| Thermal Shock Parameter, $TSP_3$, $\frac{\lambda\,MOR(1-\nu)}{E\,\alpha_T}$ | 15000 | 8000 | 1300 |
| Decomposition Point °C | 2300 | 1350 (melt) | 1850 (melt) |
| Safe Working Temperature unloaded, °C | < 1600 | < 1200 | < 1500 |

*Table 1 – A summary of different properties for 3 types of dense, sintered polycrystalline ceramics. It should be emphasized that these properties are for pure, dense substances, and not in the porous form encountered in filters. The three materials have mechanical and thermal properties which make them suitable for application to diesel particulate filters. All three are commercial products, SiC from Ceradyne, Inc., Cordierite from Asahi Glass Company Ltd., and Mullite from McDanel Refractory company. (11)*

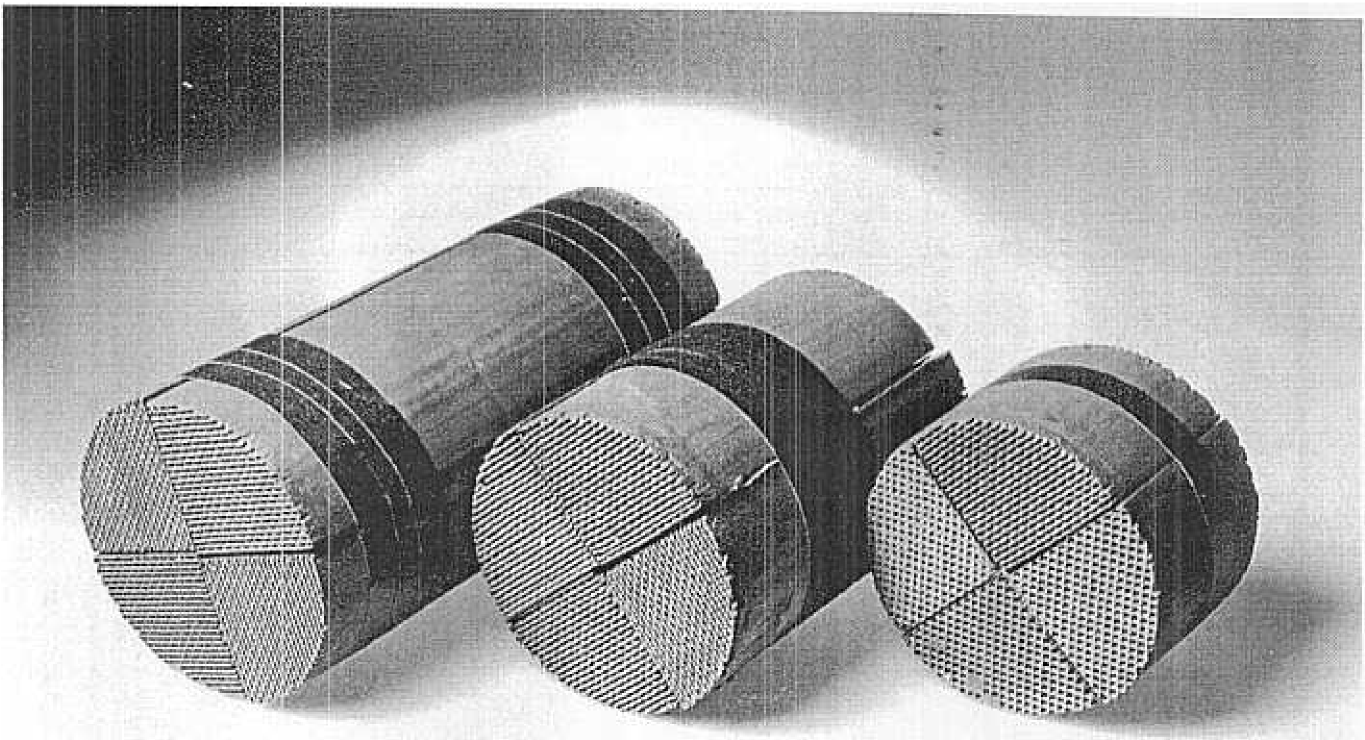

*Fig. 1 – Filter substrate manufactured out of Silicon Carbide. The diameter of the units are 170 mm and the wall thickness is 1 mm. Length 130, 275 and 380 mm.*

Another property that is advantageous for the SiC filter is a high thermal conductivity. From Table 1 it can be seen that the thermal conductivity of polycrystalline SiC is about 30 times higher than that of Cordierite, and about 20 times higher than that of Mullite. A consequence of this, as will be shown later in the paper, is a reduction in the temperature gradients, as well as the peak temperatures encountered during the regeneration process.

Not all of the properties for SiC are improved with respect to Cordierite. Table 1 shows that the thermal expansion coefficient for SiC is about five times larger than that of Cordierite, and about the same as Mullite. For a given temperature difference, then, the thermal stresses in an SiC structure would be the same amount greater than those with Cordierite and about the same as those of Mullite. A mitigating factor is found in the thermal conductivity, however, since this tends to reduce the local temperature gradients, which would lower the thermal stresses. For the overall filter structure at a uniform temperature, however, the SiC would have a higher thermal stress than the Cordierite.

A compensating factor to this is the higher strength of the SiC, which is at least twice as great as that of Mullite or Cordierite. Since it is stronger than the Cordierite, it is then able to withstand higher stresses. The compensation is indicated by the thermal shock parameter, which is a combination of the thermal expansion coefficient and the mechanical strength of a material. If the thermal conductivity is taken into account ($TSP_3$), then the SiC has a higher thermal shock parameter than Cordierite and Mullite. If it is not taken into account ($TSP_1$) then it is lower than Cordierite and about the same as Mullite. As will be shown later on in the paper, the thermal conductivity of the SiC plays a significant role in its behavior during use as a diesel particulate filter substrate. It is therefore felt that $TSP_3$ is more relevant. In the comparison of these three materials, the Mullite is the poorest choice of the three materials, since it has high thermal expansion, but a low strength.

From the point of view of the basic physical properties of SiC, then it can be seen that there are some significant potential benefits associated with the use of SiC as the substrate material for a wall flow type diesel particulate filter. The work of the authors, and that of others show that it is possible and practical to manufacture a substrate with suitable porosity and strength, to be used as a diesel filter (2,3). Reasonably conventional manufacturing techniques can be used to construct these structures. These techniques include an extrusion process, as well as high temperature processing.

In terms of cost, the basic raw material used in the filters is commercial grinding grain SiC, which is relatively cheap. The extrusion techniques used are no more expensive than those involved with the production of substrates out of Cordierite or other ceramics. Since much higher temperatures are involved with the high temperature processing, however, processing costs will be higher than for other materials. Extruder wear may also be increased because of the abrasiveness of SiC. Since currently only prototype filters are being produced, one cannot say with certainty what this additional cost will be in a practical production system.

Figure one shows photographs of a substrate material made out of SiC which has been manufactured using these techniques. They are currently limited by the size limitation of the extruding equipment. The pie shape sections can be conveniently grouped to make larger structures. Reference (2) has shown that the use of smaller elements combined to make a larger unit gives some structural advantages. The authors' experience to date has not indicated cracking problems with the configuration shown in Figure one.

Because of the good electrical properties SiC ceramics are used as the basic material for making heating elements. Another common application of SiC is the so-called "kiln furniture" which is a type of structural ceramic used to support e.g. porcelain or Cordierite during sintering. Here it is possible to take advantage of the very high strength at high temperatures, the good thermal cycling properties and good oxidation resistance (can be used up to 1600°C in air continuously).

The last mentioned application of SiC as a structural ceramic for high temperatures with thermal cycling lead among other things to the conclusion, that it would be suitable as a substrate for diesel particulate filters.

## PROPERTIES OF POROUS SUBSTRATES

The previous material has indicated that on the basis of the properties of pure polycrystalline material, SiC is a very good candidate for use as a diesel particulate filter substrate material. However, due to forming and processing techniques, the properties of porous filter materials are not expected to be the same as those for pure, solid materials. Therefore, a series of experiments have been performed to compare the relevant properties of SiC to Cordierite. Mullite was not included in the comparison, since the solid phase properties of the material were not judged to be significantly attractive.

**FLOW PROPERTIES** – An ideal particulate filter has zero pressure drop and a filtration efficiency of 100%. This is not obtainable in practice, of course, but intensive development work on the SiC filter substrates has given promising results such as a very low pressure drop and good trapping efficiency in the same filter.

The honeycomb type wall-flow-filters (WFF) have a larger filtering surface per unit of volume than depth filters (foam filters, woven fiber fabric and pressed, sintered fibrous substrate). This advantage is important when the relatively limited space on a vehicle is taken into account. Furthermore, for a given filter volume it is not necessary to regenerate a wall-flow-filter as frequently as is necessary for a depth filter. Another advantage of the WFF's is that the filtration efficiency generally is larger, typically 80-90% compared to 40-60% for a foam filter (6) – and the efficiency is not sensitive to exhaust gas flow variations. A problem with depth filters is "blow off", where the trapped soot is blown off the filter when the gas velocity through the filter is too high.

Generally the factors that increase the filtration efficiency such as reduced mean pore size, larger wall thickness and reduced porosity will exhibit a negative influence on the pressure drop, thus giving higher pressure drop (or back pressure) leading to increased fuel consumption of the vehicle.

Theoretical Considerations – The permeability for a filter substrate is a measure for what the pressure drop will be at a given gas flow rate. That is, the higher the permeability, the lower the pressure drop at a fixed flow.

For a given substrate an increase in wall thickness, gas flow rate and/or in gas viscosity will increase the pressure drop over the filter. These characteristics are seen for flow in the laminar region from Darcy's law:

$$\Delta P = \frac{t \cdot \eta \cdot V}{\alpha} \qquad (1)$$

where: $\Delta P$ is the pressure drop, t is the filter thickness, $\eta$ is the dynamic gas viscosity, V is the linear gas flow rate and $\alpha$ is the permeability.

If the mean pore size is reduced, the permeability of the filter substrate will be reduced hence leading to higher pressure drop. This is concluded by Kitagawa (12) and can also be concluded from an empirical formula (13).

$$\alpha = \frac{\varepsilon^3 \cdot (\Phi \cdot D)^2}{150 \cdot (1-\varepsilon)^2} \qquad (2)$$

where: $\varepsilon$ is the fractional porosity, $\Phi$ is the sphericity of the substrate grains (defined as the surface of the sphere with the same volume as the grain divided by the surface of the grain i.e. $\Phi < 1$), and D is the grain diameter.

Kitagawa (12) proposes that it is preferable to have a very narrow distribution of pore sizes in order to get the best possible filtration efficiency at a given pressure drop. A wide distribution of pore sizes with both very large pores and very small pores will give a relatively low efficiency since the soot particles will "slip" through the larger pores.

It is very difficult to obtain a narrow pore size distribution in an oxide ceramic like Cordierite, since the pore forming material shrinks and since grain growth during

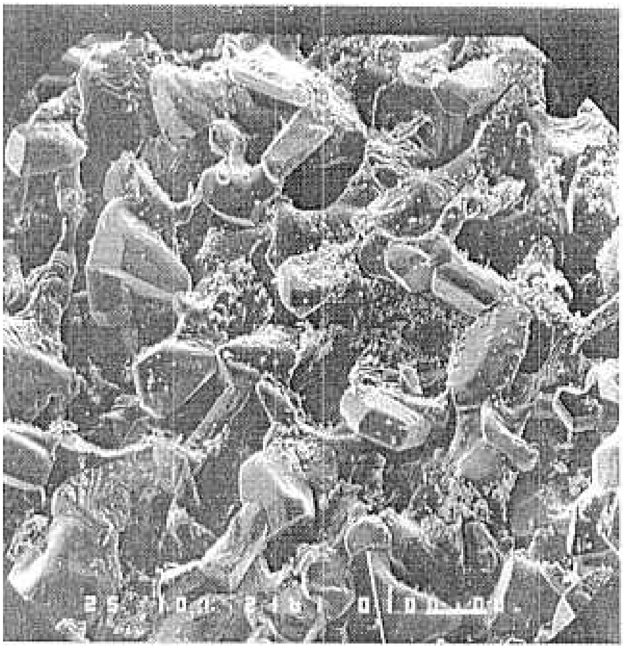

*Fig. 2 – A Scanning Electron Microscope picture of the structure of an SiC based substrate for a diesel particulate filter. The photograph is magnified by a factor of 100.*

sintering is difficult to control (2). In the SiC filter substrate there is no such problem because of a different sintering mechanism, i.e. the distribution of the pore size is very narrow because of a very narrow distribution of grain sizes in the substrate. Another advantage in the SiC substrate is that there is no closed porosity and the pores have a capillary structure. Figure two shows a SEM photograph of a typical micro structure.

The permeability is very much dependent on the porosity of the filter substrate. The porosity of the present SiC substrate is 50% which gives a good combination of mechanical, flow and filtration properties. This value is therefore chosen as a fixed value for different pore sizes, which can – at least to some degree – be chosen freely. These qualities give the possibility of obtaining both a high filtration efficiency and a very low pressure loss through the design of the filter substrate.

Experimental Results – The above mentioned advantages of the present SiC filter substrate become quite clear when it is compared to a typical Cordierite filter substrate. Flow tests were made both on thin disks of the two types of material and on whole filters without soot load. The permeability for the two different types of filter substrate was obtained from the measurements on the thin disks. The pore size of the SiC material was 40μ and that of the Cordierite slightly smaller at 33μ. It was found that the SiC filter substrate has a permeability about five times as large as that obtained for Cordierite substrates with the same filtration efficiency.

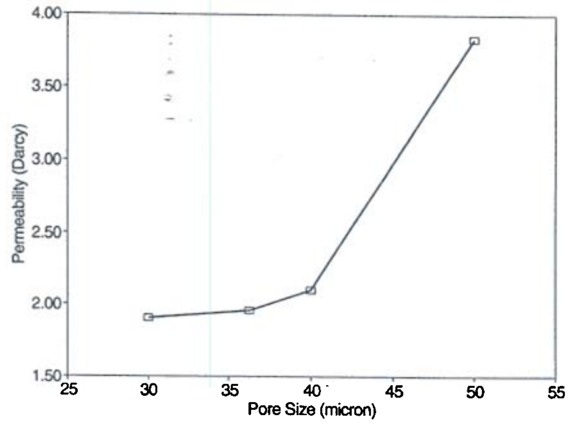


*Fig. 3 – Permeability as a function of mean pore size for SiC filter substrate material.*

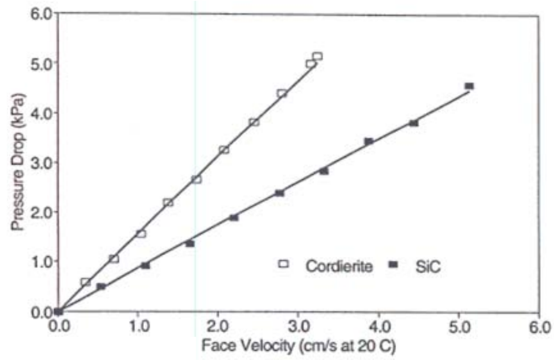


*Fig. 4 – Filter pressure drop as a function of filter face velocity for a Cordierite filter and an SiC filter substrate.*

The permeabilities obtained were:

Cordierite (33μ pores): $4.3 \times 10^{-13}\ m^2 = 0.43$ Darcy

SiC (40μ pores): $2.1 \times 10^{-12}\ m^2 = 2.1$ Darcy

Figure three shows how the permeability varies with mean pore size for the SiC substrate and it is seen that the permeability has approximately a parabolic relationship with pore size, as indicated in Equation (2) above.

When the pressure drop was measured on clean, whole filters of the same materials, the tendency was the same although the difference in pressure drop was not as large as might immediately be expected from the permeabilities alone. This can be seen from Figure four.

The reason for this is that the wall thickness of the SiC filter is 1 mm while it is 0.43 mm for the Cordierite filter and that the cell density is 8 cells per cm2 for the SiC substrate and 16 cells per $cm^2$ for the Cordierite substrate. For the measured permeabilities and wall thicknesses, it is expected that the pressure drop through the Cordierite would be 2.1 time greater than through the SiC, in good agreement with the measured difference of 1.8. Engine dynamometer test showed a similar relationship between the two types of filter substrate for a gas temperature of 250°C.

For filtration efficiencies of about 75 to 85%, the smaller pressure drop (or back pressure) for the SiC substrate compared to that of the Cordierite substrate is clearly an advantage, since the increase in fuel consumption of the vehicle will be lower while the filtration efficiency is the same. This gives the possibility of increasing the filtration time and thus increasing the time between filter regenerations.

**MECHANICAL PROPERTIES** – Ceramics are brittle materials and because of the low ductility, the compressive (crush) strength is greater than the tensile- and bending strengths (flexural strength or Modulus Of Rupture, MOR). This is a consequence of the fact that the strength of the ceramic materials is limited by the largest defect in the structure. For porous materials such as filter substrates, this means that the largest pore limits the strength and the importance of a narrow pore size distribution thus becomes quite clear. In more general terms, the strength and stiffness of the ceramic material will decrease as porosity and pore size increase.

SiC has long been known to have excellent mechanical properties at low as well as at high temperatures. The room temperature flexural strength of dense sintered SiC has been reported as high as 720 MPa (14), but a flexural strength of about 400 MPa is more typical (see Table 1). As the matter of fact, the strength of the SiC increases slightly with increasing temperature (15). This fact and the good thermal properties and corrosion resistance make SiC a suitable material for many high temperature applications.

Strength measurements – The mechanical properties of the porous SiC were investigated in order to evaluate the suitability as a diesel particulate filter substrate. A material was chosen which simultaneously exhibits a high filtration efficiency and a low back pressure. This material has a porosity of 50% and a mean pore size of 40μ. It consists of an extruded honeycomb structure with a wall thickness of 1 mm and a cell size of 2.5 × 2.5 mm.

The following strengths were measured: $MOR_A$, $MOR_B$, $MOR_C$, A-Crush and B-Crush. Figure five shows the samples which were tested and the orientations involved for the different stresses. The MOR values were measured by 3 point bending and the crush strengths were measured using a hydraulic press.

When the strength of brittle materials such as SiC and Cordierite are evaluated, it is necessary to use the so called Weibull statistics (14). For a component of a given size, surface finish, and microstructure the probability of failure, S, when a stress, σ, is applied is given by:

$$S = 1 - \exp\{-(\sigma/\sigma_o)^m \qquad (3)$$

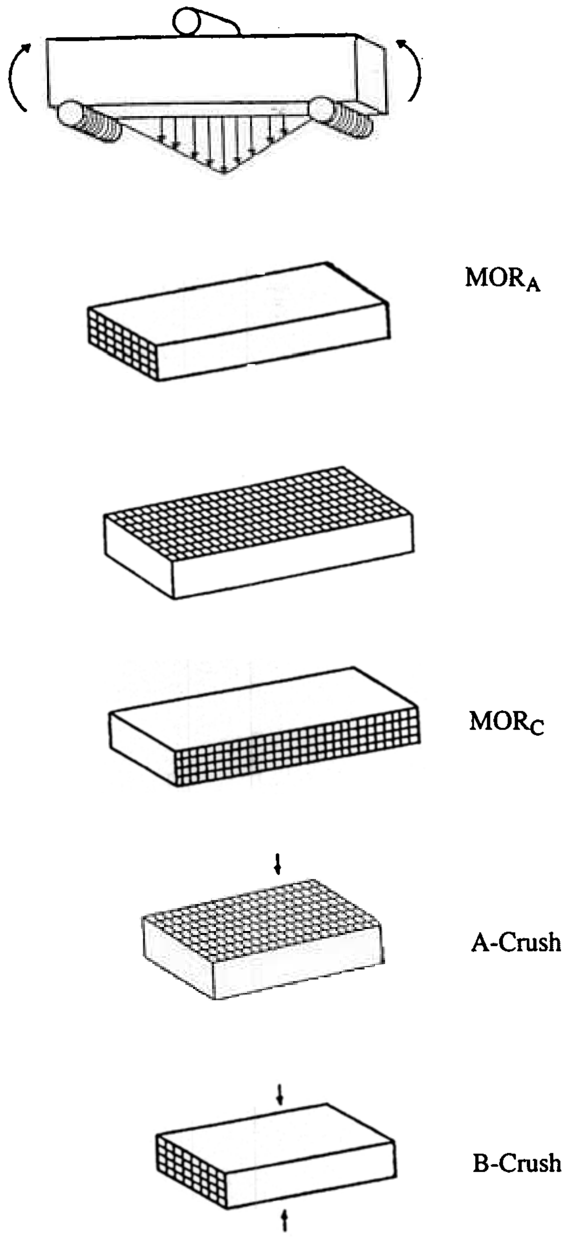


*Fig. 5 – Orientations of strength test samples for determination of MOR and crush strenght.*

where: $\sigma_0$ is a normalizing factor and m is a material constant, the Weibull modulus.

The strength of the material is then given by the stress where the probability of failure is 50%.

The equation above can be altered to:

$$\ln(\ln(1/(1-S)) = m \ln(\sigma/\sigma_o) \qquad (4)$$

When the above equation is plotted, it will give a straight line with slope m. This type of plot is called a Weibull plot.

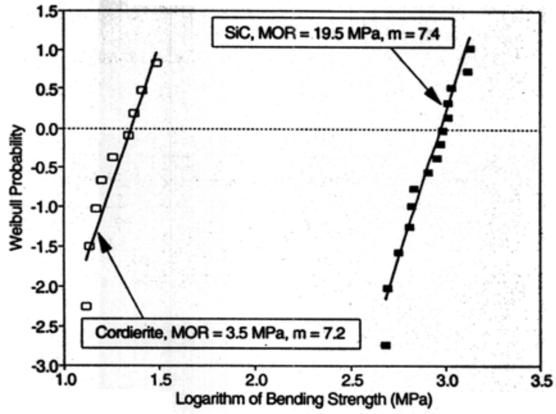


*Fig. 6 – Weibull plots of $MOR_A$ for SiC and Cordierite.*

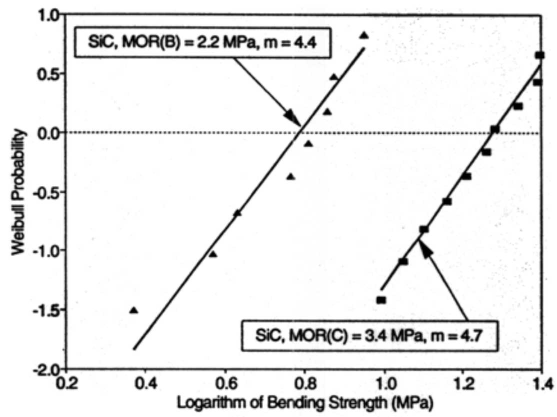


*Fig. 7 – Weibull plots of $MOR_B$ and $MOR_C$ for SiC.*

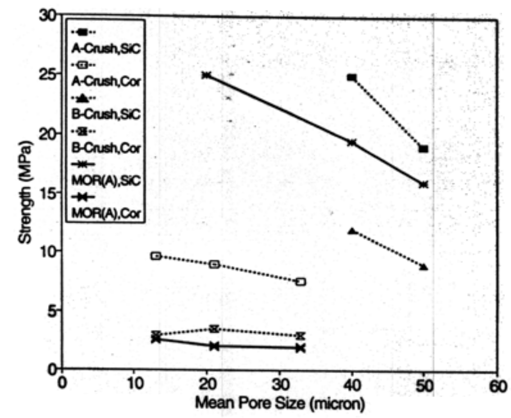


*Fig. 8 – A comparison of the MOR and crush strength for SiC and Cordierite as a function of mean pore size.*

*Fig. 9 – A Scanning Electron Microscope picture of a fracture in the SiC substrate structure. Note that the fractures occur in the grains, and not necessarily at the grain boundaries. The photograph is magnified by a factor of 300.*

The probability for failure for the i'th ranked sample in a group of N is found from:

$$S_i = i/(i+N) \qquad (5)$$

A typical value for the Weibull modulus is between 5 and 10 for dense sintered ceramics. For porous filter substrates the value normally will be lower.

Experimental results – The Weibull plots for $MOR_A$ for SiC and Cordierite are given in Figure six and the $MOR_B$ and $MOR_C$ for SiC are given in Figure seven.

For the purpose of comparison, the $MOR_A$ was measured for both SiC and Cordierite. The strengths for the other values for Cordierite was taken from Gulati and Sherwood (16). The tested Cordierite has a porosity of 46% and a mean pore size of 21μm.

The Weibull modulus for all samples is generally higher than expected (m = 7.5 for $MOR_A$ for both SiC and Cordierite and m = 4.5 for $MOR_B$ and $MOR_C$ for SiC). This is a good result since it is an indication that the materials are homogenous in structure.

The obtained strengths for SiC are up to 5 times greater than those of Cordierite.

This was also to be expected from the values for the dense materials (Table 1). The higher strength of SiC makes it more resistant to the mechanical stresses and vibrations to which a diesel particulate filter will inevitably be exposed. It also makes the SiC material more resistant to thermal stresses during filtration and regeneration even though SiC has a larger elasticity modulus than Cordierite.

To illustrate how the pore size influences the strength, the values for $MOR_A$, A-Crush, and B-Crush are plotted against mean pore size for SiC and Cordierite in Figure eight. The porosity of the SiC samples is 50% and the porosity of the Cordierite (16) is 50%, 46% and 43% for the 3 different pore sizes, 13µm, 21µm and 33µm respectively. The data for Cordierite are taken from Gulati (16).

Figure eight shows that the strength of the SiC is generally much larger than for Cordierite independent of pore size. The high strength of the SiC filter substrate is due to very good contact between the individual ceramic grains in the material. This is seen by examining the fracture surface. Figure nine shows a SEM photomicrograph of the fracture through a SiC sample with 50µm pores. This type of fracture – transgranular – is typical for the SiC material and is an indication of a strong grain boundary in that it is the material itself which breaks and not the bond between grains.

**THERMAL CONDUCTIVITY** – For the application as substrate for a diesel particulate filter, it is necessary that the material used is able to withstand high temperatures, thermal cycling, and severe thermal shocks. It is well known that both SiC and Cordierite have excellent resistance to thermal shock, and since these two materials have other desirable properties they are therefore suitable for the application.

Maximum Temperature – Cordierite is an excellent material for diesel particulate filters. It has, however, one big disadvantage which has been widely described in the literature: The substrate melts down and/or cracks if the regeneration of the filter is not performed carefully under intensive control of flow, temperature etc. This is due to the very low thermal conductivity and low melting temperature of the Cordierite. The thermal conductivity of Cordierite is on the order of 1-2 W/m-°C for pure dense material as compared to about 100 for the SiC. This makes the filter sensitive to both a maximum soot load and an uneven distribution of the soot. With electrical regeneration, a high soot load (more than 4 grams /liter filter) (17) on a 12.5 l Cordierite filter makes it necessary to regenerate the filter very slowly (3-8 hrs) with a very well defined air flow. This can be acceptable for a vehicle which can be put in a garage over night for regeneration (i.e. a city bus) but it is clearly a problem for vehicles used continuously, or for on-road vehicles driven over long periods of time.

Normally, the temperature during filtration and regeneration must be kept below 1000-1100°C for a Cordierite filter. Higher temperatures and/or large temperature gradients will lead to either cracks or to local melt down which again will lead to a large drop in filtration efficiency. When this occurs, the filter must be replaced.

These problems are not seen in the SiC filter substrate because of its high thermal conductivity which has been measured to be 11 W/m-°C at 60°C with 50% porosity and high service temperature (1600°C). During regeneration, the energy related will be evenly distributed throughout the ceramic due to the high thermal conductivity, hence thermal stresses are suppressed and a high durability is achieved. Even with short term and severe regeneration no local temperature peaks like those in Cordierite (12) are observed. The upper limit for soot load and the distribution of the soot in the SiC substrate is thus not limited by the thermal properties. The "working window" (or flexibility) during filtration and regeneration is therefore larger for SiC than for other substrates.

The high thermal conductivity of the SiC will also make it easier to have a uniform distribution of temperatures in the filter when it is heated (electrically or with a burner) prior to the regeneration. Hence thermal stresses caused by large thermal gradients in the filter from an uneven combustion of the soot are unlikely.

Thermal Shock – The resistance to thermal shock for a material can be quantified empirically through strength, stiffness, thermal expansion and thermal conductivity in the so called Thermal Shock Parameter (TSP). This parameter can be calculated in at least 3 different ways (15, 16, 18)

$$TSP_1 = \frac{MOR}{E \cdot \alpha_T \cdot \Delta T} \qquad (6)$$

$$TSP_2 = \frac{MOR \cdot (1-\nu)}{E \cdot \alpha_T \cdot \Delta T} \qquad (7)$$

$$TSP_3 = \frac{\lambda \cdot MOR \cdot (1-\nu)}{E \cdot \alpha_T} \qquad (8)$$

In these formulas, $\lambda$ is Poisson's ratio, E is the elasticity modulus, $\alpha_T$ is the thermal expansion coefficient, $\lambda$ is the thermal conductivity, and $\Delta T$ is the temperature change of the sample. (In table two, $\Delta T$ = 1000°C – 25°C)

Depending on which formula is used to calculate the TSP, either Cordierite or SiC can be shown to have the greater value. The higher the value of the TSP, the better the thermal shock resistance. Since the mechanical and thermal properties depend on the orientation of the material, the TSP's are given in intervals in two.

| Material | SiC | Cordierite |
|---|---|---|
| $TSP_1$ | 0.009 - 0.05 | 0.21 - 0.72 |
| $TSP_2$ | 0.008 - 0.04 | 0.15 - 0.54 |
| $TSP_3$ | 30-460 | 74-259 |

*Table 2 – The different thermal shock parameters for porous SiC and Cordierite.*

Table two shows that when the thermal conductivity is taken into account ($TSP_3$), the TSP's for SiC and for Cordierite are of comparable magnitude. The conclusion from these empirical values is, that Cordierite is somewhat

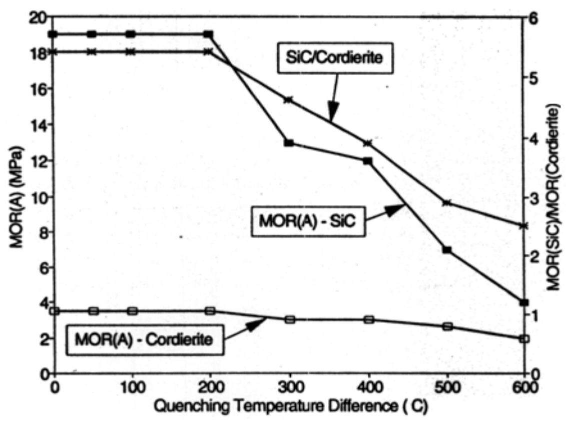


*Fig. 10 – Bending strength of samples of SiC and Cordierite quenched in 20°C water for different quenching temperatures. The ratio of the two strengths is also shown.*

better to withstand thermal shock than SiC. It should, however, be taken into account that the thermal conductivity of the SiC material is so high that thermal gradients are much smaller with SiC. This can be seen from the regeneration tests.

In order to test the thermal shock characteristics, small samples (1 × 2 × 7 cm ) were cut from filters of SiC and Cordierite. These samples were annealed in air for 20 minutes at temperatures from 70 to 620°C and were then quenched in room temperature water. After this rather severe treatment the 3 point bending strength ($MOR_A$) was measured for all the samples (6 samples of each material at each temperature. These strengths are plotted against quenching temperature in Figure 10. In addition, the ratio of the strength of SiC to that of Cordierite is also shown.

The figure shows that both materials begin to be affected by the quenching process at temperatures of 200-300°C. The relative strength of the cordierite decreases more rapidly than that of the cordierite. For the Cordierite, the strength after quenching from 620°C to 20°C is reduced by 43%, while for the SiC, the reduction is 74%. In terms of absolute strength, the $MOR_A$ of the SiC is 2.4 times higher than that of the Cordierite when both quenched from 620°C to 20°C.

Quenching from 220°C to room temperature in less than a second does not affect the strength of either material even though this is an extremely severe treatment. The results for SiC are in very good agreement with the results of Koumoto for 20 to 30% porous fine grained SiC (19). A summary of the thermal and physical properties of the two porous substrate materials SiC and Cordierite is given in three.

## HIGH TEMPERATURE CORROSION OF SIC BASED DIESEL FILTERS

One possible concern of the use of SiC as a diesel particulate substrate may be that of high temperature corrosion. The authors have not yet observed any noticeable effects of this process, but since data are not yet available for very long term testing, it is thought that the process which could lead to corrosion should be clarified.

SiC oxidizes relatively easily in connection with atmospheric air according to the reaction [1], thereby forming a tight layer of amorphous silica on the surface of the material.

$$SiC + O_2 \rightarrow SiO_2 + CO_2 \qquad [1]$$

At high temperatures, (about 1400°C) this layer recrystallizes to form cristobalite. Further oxidation of the SiC below this layer can then only occur through diffusion of oxygen through the reaction layer. The rate of oxidation occurs according to a parabolic "power law" and is temperature dependent according to the Arrhenius kinetic equation. The activation energy, $E_a$, in pure oxygen has been determined to be 84.6 kJ/mol for amorphous silica and 65.3 kJ/mol for cristobalite (21). At 900°C in pure oxygen, a 40µm grain will be oxidized to a depth of < 0.05µm. This corresponds to a reduction of the contact area (SiC-SiC) between the sintered grains of approximately 0.2%. At 1500°C, the reduction will be about 1%. For extreme temperature excursions, the layer can crack and therefore allow local free passage of oxygen to the SiC, and a consequent increase in the rate of oxidation.

The presence of water vapor (50% rh at 20°C) and/or CO% in the corroding gas accelerates the process significantly (22). The rate of corrosion at 900°C in a "wet" atmosphere is equal to the rate of corrosion at 1500°C in pure dry oxygen. This is caused by a shift in the corrosion mechanism from the pacifying oxidation discussed above to an active oxidation in which the following reactions are thought to participate:

$$SiC + 2O_2 \rightarrow SiO(g) + CO \qquad [2]$$

$$SiC + 4H_2O \rightarrow SiO_2 + CO_2 + 4H_2 \qquad [3]$$

$$SiO_2 + H_2 \rightarrow SiO(g) + H_2O \qquad [4]$$

The reaction products are gaseous. Gasses such as $SO_2$, $CH_4$, and $H_2S$ will have the same effect.

The worst conceivable case is the deposit of large amounts of combustion products, ash, which could form an alkaline melt on the surface of the SiC grains. The protective layer of $SiO_2$ is easily soluble in the alkaline melt with a rate of diffusion for oxygen through the deposit to the SiC surface which is about $10^4$ times greater than through solid

$SiO_2$. The process is now referred to as accelerated corrosion (22). The content of alkaline sulfates and carbonates in the ash will therefore be very important for the use of SiC based filter material. At 900°C for example with free access to $Na_2SO_4$ deposits, the corrosion rate will correspond to a reduction in the grain size of 50 μm/hr, which corresponds to a complete melting of the material.

An evaluation of the importance of the corrosion processes for the life time of the filters must therefore be conducted on the basis of the expected operating conditions with respect to the combination of temperature, time, and atmospheric composition. The operating conditions can be divided up into two periods: particle accumulation and controlled regeneration. In addition to this is the worst case

| **Property of Filter Substrate** | **Silicon Carbide SiC** | **Cordierite $2MgO2Al_2O_35SiO_2$** |
|---|---|---|
| Bulk density, ρ, $g/cm^3$ | 1.6 | 1 |
| Porosity, ε,% | 50 | 46 - (16) |
| Cells per $cm^2$ | 8 | 16 |
| Cell size, mm | $2.5 \times 2.5$ | $2.1 \times 2.1$ |
| Wall thickness, t, mm | 1 | 0.43 |
| Filter surface per liter $m^2/l$ | 0.42 | 0.63 |
| Mean pore size, μm | 40 | 33 - (16) |
| Filtration efficiency,% | 75 - 90 | 75 - 85 - (16) |
| Permeability, α, Darcy | 2.1 | 0.43 |
| Back pressure, mbar (velocity = 2cm/s, clean) | 20 | 35 |
| Modulus of Elasticity, E, GPa | 85 | 5 - (16) |
| Poisson's ratio, ν - (11) | 0.16 | 0.26 |
| Compressive strength, longitudinal, $\sigma_A$, MPa | 25 | 9 - (16) |
| Compressive strength, transverse, $\sigma_B$, MPa | 12 | 3.4 - (16) |
| Bending strength, $MOR_A$, MPa | 19.5 | 3.5 |
| Bending strength, $MOR_B$, MPa | 2.2 | --- |
| Bending strength, $MOR_C$, MPa | 3.4 | 1 - (16) |
| Electrical resistivity, Ω cm | 1 | $> 10^{-11}$ |
| Specific heat, $C_p$, J/kg-°C, at 25°C - (20) | 750 | 600 |
| Thermal conductivity, λ, W/m-°C, at 25°C | 11 | < 0.5 |
| Thermal conductivity, λ, W/m-°C, at 630°C | 7 | < 0.5 |
| Thermal expansion coefficient, $\alpha_T$, 1/°C | $4.6 \times 10^{-6}$ | $1.0 \times 10^{-6}$ |
| Thermal Shock Parameter, $TSP_3$, $\frac{\lambda \times MOR \times (1-\nu)}{E \times \alpha_T}$ | 150 - 850 | 150 -750 |
| Decomposition/Melting temperature, °C | 2300 | ~ 1200 |
| Critical regeneration temperature limit °C | ~ 1600 | ~ 1000 |
| Max. safe loading, g/liter filter | 20 [back pressure] | 7 [thermal load] - (17) |
| Max. observed temperature during "Worst Case" regeneration, °C | ~ 900 | > 1400 |

*Table 3 – Properties for two types of ceramic substrates as used in a wall flow filter. Where no reference is given, the properties have been measured by the authors.*

situation which results from a failure of the regeneration control system. During normal operation and accumulation of soot, the temperature will not exceed 600°C and the filter will remain completely unaffected. For slow regeneration processes, the temperature will remain lower than 800°C. During a rapid controlled regeneration, the temperature will only increase to about 900°C depending on the amount of accumulated particles. The filter will be exposed to this temperature for a very short period of time, approximately 30 seconds, after which the temperature falls. For filter usage during continuous driving it will be necessary to regenerate about 3-4 times a day. For a filter lifetime of about 2 years, this corresponds to a life cycle regeneration time of 20-30 hours at 900°C. Excluding corrosion from alkaline ash deposits, the corrosion will be minimal. For the worst scenario, where large amounts of particulate matter are accumulated, experience with Cordierite filters shows a short term increase in temperature to over 1200°C. Corrosion could be significant but not catastrophic under these conditions, and the life of the filter will depend upon how often these conditions are encountered. However, results to date with regeneration of SiC filters indicate that the temperatures are much lower, nearly always lower than 900°C.

The problem with alkaline melt becomes of importance for temperatures about the eutectic temperature for a given mixture of alkali sulfates, carbonates, chlorides, etc. This temperature can be as low as 300-400°C. How much damage, if any, might occur will depend on the quantity and composition of the deposit/ash from the fuel and particulate. A point of interest in the future evaluation of long term durability of SiC filters is then the composition of the lubricant additives, since they may remain on the filter after regeneration.

A recent study of the effects of lubricating oil and fuel composition on ash accumulation has shown that after 3000 hours of operation with conventional fuels and lubricants in a 240 kW engine, 1.27 kg of ash were accumulated in a particulate filter (23). The ash contained a variety of metal, some of which might give long term difficulties. By switching to low sulfur fuel and ashless lubricant, the amount of ash accumulated was reduced to 0.06 kg, and the elements which might cause a long term corrosion problem were not present. On the contrary, a large portion of silicon was observed in the deposits, which certainly presents no problem for the filter.

It can then be stated that the general demand for all types of filters, that low ash lubricant and low sulfur fuel be used, will contribute to a reduction or elimination of corrosion potential on SiC substrates.

## ENGINE TEST RESULTS

In all the engine tests reported in this section, the filters were constructed of material corresponding to the characteristics given in two. They were constructed by sealing 4 quadrants of the type shown in Figure 1 into a round cylindrical unit. The volume of the filters were adjusted by changing the length of the unit.

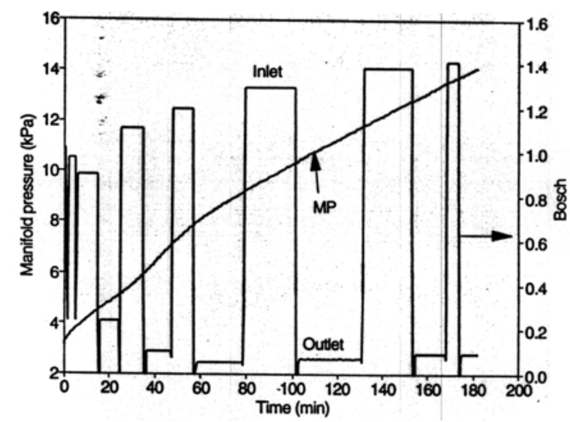


*Fig. 11 – Bosch smoke number at the inlet and outlet of an SiC filter, and increase in back pressure during steady state accumulation of particulate matter.*

**FILTRATION** – Figure 11 shows the filtration effects for a clean filter on a 9 liter turbocharged diesel engine. The engine was run at a constant speed and load, and particulate matter accumulated. The effect of the filter was monitored by comparing the Bosch smoke number at the inlet and outlet of the filter. The tests were started with a clean filter. The Bosch smoke number initially was 0.95 at the filter inlet and about 0.25 at the outlet of the filter. After one hour the inlet Bosch number increased to 1.25 due to the increasing back pressure, while the smoke number out of the filter decreased to under 0.1. This indicates the increasing efficiency due to the increase in the mass of particles on the filter. While the Bosch number cannot be translated directly into a particle collection efficiency, the numbers shown are consistent with a trapping efficiency in the range of 70 to 90%.

**REGENERATION** –

Exhaust gas driven regeneration – SiC particulate filters have been constructed and tested for a variety of engine types and regeneration strategies. Figure 12 shows engine back pressure, speed and load, and filter temperatures for regeneration of an uncoated SiC filter. Although the authors do not have experience with catalytically coated SiC substrates, it is believed that it is technically possible to apply a catalytic coating. Specifications for the engine and filter used for the test are shown in Tables 4 and 5 on the next page. Particles are accumulated on the filter at a medium load, and then the regeneration is started by the high exhaust temperature resulting from increasing the speed and load of the engine. No auxiliary regeneration energy was used. When the filter inlet reaches about 550°C, the regeneration starts, as can be seen by the reduction in the back pressure in the exhaust manifold.

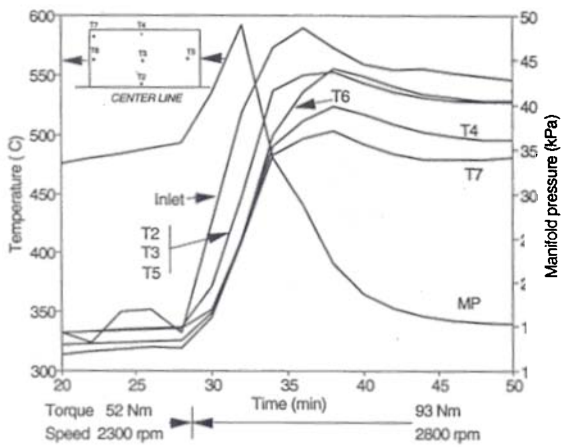


*Fig. 12 – Filter temperature distribution and exhaust back pressure during the exhaust temperature driven regeneration of an SiC filter under steady state conditions. Engine and filter specifications are given in Tables 4 and 5.*

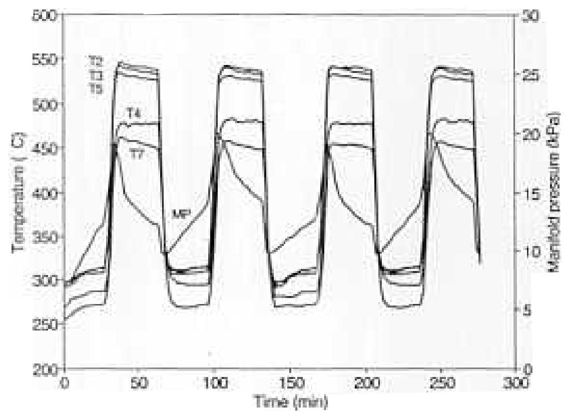


*Fig. 13 – A sequence of regenerations for the conditions shown in Figure 11.*

With the engine maintained at constant speed and load, it can be seen that the regeneration proceeds in a controlled fashion, over a period of about 10 minutes, with a very uniform temperature distribution within the filter. The filter temperatures are low, and in this case do not exceed 600°C anywhere during the entire course of the regeneration. Figure 13 shows a sequence of 4 of these tests conducted in series. It can be seen that the back pressure drops to essentially its initial value, confirming the completeness of the regeneration. The uniformity of temperature is observed in all the cycles.

"Worst Case" Regenerations – In order to demonstrate the additional margin of safety provided by the use of SiC substrate material, experiments were performed which would be expected to lead to the thermal destruction of other types of filters. The basic concept is to start a regeneration, and then put the engine into a condition which supplies

| | |
|---|---|
| Engine displacement - liter | 1.6 |
| Bore - mm | 75.5 |
| Stroke - mm | 86.4 |
| Cylinders | 4 |
| Combustion System | Indirect Injection |
| Intake System | Normally Aspirated |
| Maximum Power - kW | 35 |
| Rated Speed - rpm | 4000 |

*Table 4 – Engine Specifications for the engine regeneration tests shown in Figures 12-15.*

| Filter Property | SiC | Cordierite |
|---|---|---|
| Overall length - mm | 84 or 125 | 150 |
| Overall diameter - mm | 175 | 145 |
| Total Filter Volume - liter | 2 or 3 | 2 |
| Cells per $cm^2$ | 8 | 16 |
| Wall thickness - mm | 1.0 | 0.43 |
| Porosity -% | 50 | 46 |
| Surface area per liter - $m^2/l$ | 0.42 | 0.63 |
| Total Filter Area - $m^2$ | 0.82 or 1.23 | 1.26 |
| Coating | none | none |

*Table 5 – Filter specifications for the filter regeneration tests shown in Figures 12-15.*

sufficient air to maintain the regeneration, but is marginal in terms of energy removal.

Figure 14 illustrates one of these tests. In this case, an uncoated SiC filter with the 2 liter capacity and the same characteristics as the filter of Table 5 was compared to the Cordierite filter shown in Table 5. The filters were loaded at a medium speed and load, then heated by an increase in engine speed and load, and when the regeneration starts, the engine was rapidly put into idle.

Figure 14 shows that the back pressure development during the entire test was essentially the same for the two filters. This is due to the small size of the SiC filter, and indicates that the accumulation and regeneration proceeded in essentially the same manner for both filters. However, a marked difference was noted in the temperature development. For the Cordierite filter, the temperature at the center of the filter increased dramatically from the initial temperature of 680°C to a maximum in excess of 1100°C. This temperature is very near the melting point of the Cordierite, and in fact this is a typical failure mode for the Cordierite filter, in that a regeneration is started and the engine flow is

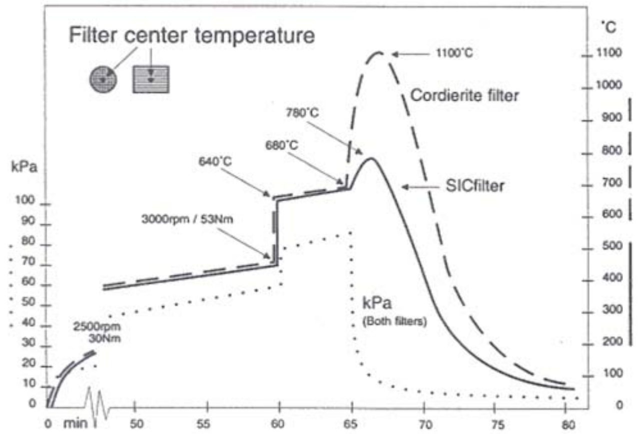


*Fig. 14 – "Worst Case" Regeneration of an uncoated SiC filter in engine exhaust gasses. The filter is loaded at a medium speed and load, then heated by an increase in engine speed and load, and when the regeneration starts, the engine is rapidly put into idle.*

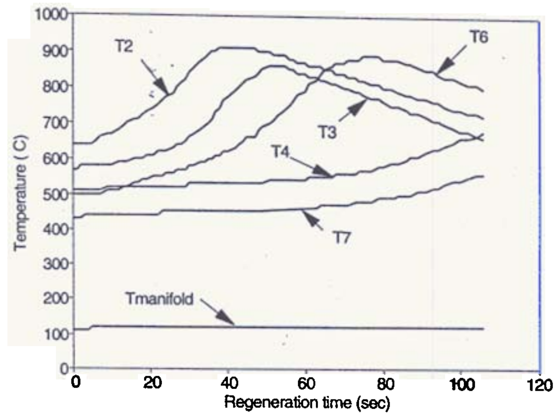


*Fig. 15 – Filter temperature distribution during a "worst case" regeneration of an SiC filter. The filter was heavily loaded and regeneration occurred during idling conditions. Engine and filter specifications are given in Tables 4 and 5. The temperature locations are the same as in Figure 12.*

drastically reduced. A Cordierite filter with a higher particulate loading would have melted in the center. Due to the low thermal conductivity, the local temperature becomes very high at the location where the regeneration front is found (24).

For the SiC filter however, the center temperature of the filter does not exceed 800°C, even though the regeneration proceeds in an equivalent manner to that of the Cordierite filter. Thus, the advantages of the use of SiC as a filter substrate are clearly demonstrated. Not only is the melting point of the SiC much higher than that of the Cordierite, its use reduces the maximum filter temperature encountered under a regeneration, further increasing the margin of safety.

An additional "worst case" type of regeneration was also investigated with this engine and a 3 liter SiC filter described in Tables 4 and 5. In this case, an uncoated SiC filter was loaded with approximately 40 grams of particulate matter at an intermediate speed and load. At this loading, a significant deterioration in engine performance was observed. Following this, the engine was stopped, and the filter was electrically heated to a temperature of about 600°C. The engine was then started and idled at 1100 rpm, at which time a very vigorous regeneration was observed. The temperature development throughout the filter during this rapid regeneration is shown in Figure 15. It can be seen that the filter temperature does not exceed 900°C at any time or location. It can also be observed that the temperature time histories are quite similar in nature to those generated by the simplified heat transfer model described below. No physical change could be detected in the filter.

Electrical Regeneration – The benefits of the use of SiC can also be observed during the electrical regeneration with the "City Filter" concept (10). This type of regeneration occurs with a filter which is located at a position in the exhaust gas system where regeneration conditions are never encountered. Therefore, the particulate matter is collected for a length of time and then burned while the vehicle is parked. This technique, which is quite sui for off-road and service vehicles with a reasonably fixed daily schedule, would allow regeneration under carefully controlled conditions.

The regeneration of an SiC filter by this technique is shown in Figure 16. Two 6 liter filters were used with the structural specifications given in Table 5. The regeneration is controlled by a combination of the electrical heating rate and a controlled flow of air through the system. A 6 kW electrical heater was used, and the air flow was 300 liters per minute. The heating element was located below and just upstream of the filter. The air flow through the filter was in a vertical direction from the bottom to the top.

Figure 16 shows the time development of the filter temperatures and the oxygen concentration at the outlet of the filter. As was the case with the exhaust gas regeneration process shown above, the use of SiC as a filter material results in a low maximum temperature, in this case approximately 800°C. Under similar flow conditions and loading, a 12 liter single Cordierite filter was found to melt in the center of the filter.

**EFFECT OF THERMAL CONDUCTIVITY** – In the experimental results, it has been shown that the use of SiC substrate results in lower and more uniform temperatures under regeneration. In order to verify the influence of thermal conductivity on the temperature development, a simplified mathematical model was used. In the model, it was assumed that a filter consists of a one-dimensional structure,

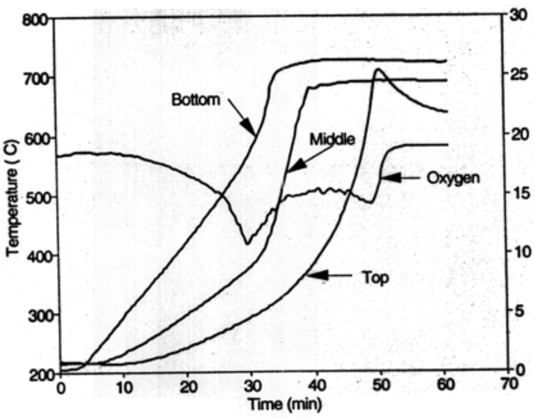


*Fig. 16 – Filter temperature and outlet oxygen concentration for an SiC filter regeneration by the use of electrical heating and controlled air flow on a parked vehicle.*

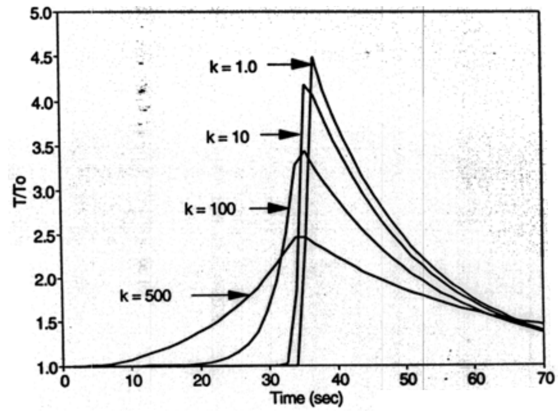


*Fig. 17 – The effect of thermal conductivity on the simulated temperature development in a simulate porous material, with space and time dependent energy release, and convective heat transfer.*

in which there is an internal energy release which is time and space dependent. In this case, it was assumed that a combustion wave, composed of a sinusoidal shape with width, δ, traverses the structure at a constant velocity, c, regardless of the substrate material. In addition, it was assumed that the combustion energy released in the material was carried away by a convective process, corresponding to flow through a porous medium.

The model results in the following partial differential equation, which was solved by conventional finite difference techniques (24):

$$\frac{\partial^2\Theta}{\partial\xi^2}+\varepsilon\frac{\pi}{2}\frac{t_o}{t_r}\frac{H_u}{C_pT_o}\sin\left[\frac{\pi L}{\delta}\left(\xi-\frac{t_o\tau}{t_r}\right)\right] =$$

$$\frac{\partial\Theta}{\partial}t+\frac{\gamma h L^2}{\lambda}(\Theta-\Theta_G) \qquad (9)$$

Where: Θ is the ratio of the temperature to the initial temperature – $T_o$, ξ is the non-dimensional distance, ε is the filter loading in grams of particle per kg of filter, $H_u$ is the heating value of the particulate matter, $C_p$ is the specific heat of the filter material, L is the length of the filter, $t_o = L^2/\alpha$ is the reference time for thermal diffusion, $t_r$ is the time for the reaction wave to move from one end of the filter to the other, ) $\tau = t/t_o$ is the dimensionless time, γ is the ratio of surface area to volume of the filter material, h is the convective heat transfer coefficient, λ is the thermal conductivity, and the subscript, G, represents the gas.

A simulation was performed for a rapid regeneration, that is a regeneration which occurs in about one minute. The combustion wave was assumed to travel at 5 cm per second over the filter length of 300mm. Typical material values were chosen based on Tables one and two. The product of the convective heat transfer coefficient and the surface area of the porous structure was arbitrarily chosen such that the temperature of the first part of the filter to be heated decreased to approximately the inlet gas temperature at the end of the energy release. The convective heat transfer was then maintained constant, and the thermal conductivity of the material was varied from 1 to 500 W/m-°C.

The results are shown in Figure 17. It can be seen that as the thermal conductivity increases, the temperature rise is much more gradual, and the magnitude of the peak temperature encountered is reduced. Similar results are also obtained with slower regenerations, provided that the convective heat transfer rates are adjusted to maintain a similar balance between energy release and removal rates. In this simple model, the effects of the convective heat and mass transfer on the reaction rate are not included. A more detailed model of the complete process, including chemical reactions, can be found in Reference (25).

The trends calculated with this simplified model are in good qualitative agreement with the experimental results for a variety of regeneration processes, both rapid and slow, for SiC filters, as shown in the results of this work and also in Reference (3). This tends to confirm the beneficial effects of a high thermal conductivity material in reducing both the maximum temperature and the thermal gradients encountered during filter regeneration.

## CONCLUSIONS

It has been shown that SiC possesses physical and thermal properties which make it well suited for use as a substrate for diesel particulate filters. When compared to Cordierite, it exhibits higher thermal conductivity, physical strength, melting temperature, working temperature, electrical conductivity, thermal expansion coefficient, and equivalent filtration efficiency. The adverse effects of the higher thermal expansion on thermal stresses are compensated by higher strength and other advantageous physical properties. Calculated thermal shock parameters indicate that SiC may be worse than or equivalent to Cordierite with regard to thermal shock. Thermal shock (quenching) experiments indicated that a substrate material of SiC has mechanical strength which is at least 2.5 times greater than that of Cordierite.

When SiC filters are regenerated under differing conditions it is found that when compared to Cordierite filters of comparable size, local temperatures are lower in the SiC filters, and that SiC filters can survive regenerations which would result in the melting of a Cordierite substrate. Long term corrosion does not appear pose a significant problem with SiC filter substrate. As with other types of filter substrates, the use of ashless lubricants and low sulfur fuels will give significant improvement in filter performance.

"Worst case" testing of SiC filters indicates that even for very severe regenerations processes, which would melt many other filter types, the maximum temperature of the SiC substrate does not exceed 900°C, and the SiC filter survives.

Simplified heat transfer calculations, which include combined effects of thermal conductivity, internal energy release, and convective removal of energy, confirm the observed advantages of a substrate with a high thermal conductivity. These advantages include lower peak temperatures and small temperature gradients during regeneration processes.

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