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HEALTH & SAFETY LABORATORY

# An Investigation Into The Potential Health Risks From Commercial Silicon Carbide Reinforced Tools.

**HSL/2005/18**

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Science Group: **Group 5**

## PRIVACY MARKING:

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# EXECUTIVE SUMMARY

This investigation, into the possible airborne fibre releases from silicon carbide and silicon nitride cutting tips used on some machine tools, was carried out for Mr. M. Nind (HSE, HIDCI3F, Sheffield). Airborne fibre levels were measured at sites using such tools and during simulated work activities in HSL's Sheffield Laboratory. The surfaces of, 'as received' and freshly fractured surfaces were examined using electron microscope techniques. Scanning electron microscope images of the freshly fractured surfaces showed that fine fibres (~ 0.2 µm diameter) were present embedded in the surface and may have the potential to become airborne. No fibres were observed at the surface of the 'as received' surfaces. Very few fibres were found on filters used to sample airborne releases either in the workplace or during laboratory simulation tests. The low levels of fibre release may be a result of the tools being worn rather than fractured. Consequently fibres, at the wear surface, are worn away and released as particles rather than fibres.

Electron microscope examination of the fibres indicated that they were very fine, often less than 0.2 µm in diameter. Therefore it is unlikely that such fibres would be detected with the conventional optical microscope techniques used to monitor airborne fibre releases.

The conclusion from this investigation is that although fine silicon carbide and silicon nitride fibres are present in these machine tools, the way that these tools are used precludes their release as fibres. In many cases the tools are subject to wetting (cutting fluids) during use and this is to be encouraged as this will further reduce the possibility of airborne fibre releases.

# 1 INTRODUCTION

Silicon Carbide / Silicon Nitride.

Machine tools with ceramic tips that include silicon carbide (SiC) or silicon nitride ($Si_3N_4$) whiskers are used for a variety of cutting operations. These SiC / $Si_3N_4$ whiskers are extremely hard[1] and exhibit long bio-persistence. If fibres released during cutting operations, become airborne and are breathed into the lung, there is the possibility of serious health affects. Mr. M. Nind (FOD Sheffield) asked HSL to determine if the use of such tips in local industry was likely to be a risk to health. To asses this risk HSL:

- Carried out on site monitoring to determine if airborne fibres were being released during the cutting processes for which these SiC and $Si_3N_4$ reinforced tips are being used.
- Used optical and electron microscope techniques to examined the tips supplied by Mr. Nind / local users of the machine tools.
- Carried out laboratory experiments to determine if fibres could be released.

Manufactures data about the tips is include in appendix 1.

[1] Silicon carbide has a hardness of 9 on Moh's scale of hardness.

# 2 ON SITE MONITORING

Mr. P. Baldwin (FSSU, Leeds) and Dr. D. Bard (IF, Sheffield) carried out airborne fibre sampling at three sites in and around Sheffield. At sites one and two, wet cutting was being carried out[2] but at site three the cutting was done dry. Samples were collected on 25 mm mixed esters of cellulose filters and the airborne fibre concentrations determined by counting in accordance to MDHS 59. Table 1 shows the volume of air sampled, the number of fibres collected, the airborne fibre concentration and the upper 95% confidence level for each sample. More comprehensive sampling information is shown on the requests for analysis sheets included in appendix 2.

The numbers of fibres meeting the MDHS 59 counting criterion were very low; from twelve samples, taken at three sites, only 51[3] fibres were counted (in 12 x 200 fields). The airborne fibre concentrations for each sample were generally below the limit of quantification (LOQ). To improve the statistical significance of the data by lowering the LOQ, data from each site was pooled[4]. By doing so it was possible to derive statistically significant airborne fibre concentrations of 0.006 and 0.005 f/ml for sites 2 and 3. However, the pooled data from site 1 (0.003 f/ml) was below the LOQ and would be reported as <0.01 f/m.

[2] In many cutting operations it is normal practice to spray the cut surface / cutting tool with a lubricant (cutting oil); this assists the cutting operation, results in a better product and prevents the release of airborne debris.

[3] Under MDHS 59 counting rules the fibres on a small proportion of the filter (200 fields of view) are counted and the total number collected and airborne fibre concentration calculated by extrapolating from this count.

[4] Sampling was carried out for different work activities using both personal and static sampling. By pooling the data statistically more robust data may be produced.

**Table 1: Measured Airborne Fibre Levels at Three Sites.**

| Site | Sample N° | Air volume (litre) | N° of fibres on the filter (in 200 fields)* | Airborne fibre concentration (f/ml)# | Upper 95% confidence limit |
|---|---|---|---|---|---|
| 1 04-0433 wet cutting | 04312/04 | 84 | 610 (2) | <0.06 (0.006) | 0.023 |
| | 04313/04 | 155 | 154 (½) | <0.04 (0.001) | 0.008 |
| | 04314/04 | 339 | 917 (3) | <0.02 (0.002) | 0.007 |
| | 04315/04 | 152 | 917 (3) | <0.04 (0.005) | 0.016 |
| | Pooled | 730 | 2,598 (8½) | <0.01 (0.003) | 0.008 |
| 2 04-0501 wet cutting | 04417/04 | 296 | 763 (2½) | <0.02 (0.002) | 0.007 |
| | 04418/04 | 1,539 | 7,181 (23½) | <0.01 (0.004) | 0.007 |
| | 04419/04 | 306 | 456 (1½) | <0.02 (0.001) | 0.006 |
| | Pooled | 2,141 | 8,400 (27½) | 0.003 | 0.006 |
| 3 04-0674 dry cutting | 04550/04 | 550 | 1,114 (3½) | <0.01 (0.002) | 0.005 |
| | 04551/04 | 100 | 307 (1) | <0.05 (0.002) | 0.015 |
| | 04552/04 | 450 | 2,141 (7) | <0.02 (0.004) | 0.009 |
| | 04553/04 | 100 | 77 (0) | <0.05 (0.001) | 0.011 |
| | 04554/04 | 100 | 1,070 (3½) | <0.05 (0.008) | 0.026 |
| | Pooled | 1300 | 4,402 (15) | 0.003 | 0.005 |

* the number of fibres found in 200 fields of view as shown in brackets () has been extrapolated to give the total number of fibres collected over the full filter area.

# The measured airborne fibre concentrations are shown in brackets alongside the reported values.

The numbers of fibres counted for the individual samples was such that the results would be reported as less than the limit of quantification.

Pooling the data from all filters (from a site) was used to increase the statistical significance and for sites 2 and 3 produced overall site airborne fibre levels above the LOQ.

# 3 LABORATORY TESTING

## 3.1 ELECTRON MICROSCOPE EXAMINATION OF SILICON CARBIDE TIPS

A number of circular cross section (1 cm diameter x 0.8 cm long) and rectangular cross section (0.5 cm square, 1.5 cm long)'ceramic tips' were received for examination by optical and electron microscopy techniques.

**Figure 1: SiC**

An optical microscope image of one of the tips is shown as figure 1. Apart from confirming the grey metallic appearance, optical microscope examination did not reveal any significant information about the material and further examination using scanning electron microscope (SEM) techniques was carried out. From this SEM examination it was possible to determine the size, appearance and distribution of silicon carbide whiskers within the tips. Energy dispersive x-ray (EDX) spectra were produced and used to determine the elemental composition of selected features (SiC fibres).

Initial examination of an 'as received' tip found that although a range of fibres were clearly visible, analysis of EDX spectra derived from these fibres indicated that few, if any, of them were silicon carbide. The main features found on the tip surface were particles of metal, principally nickel, iron and chromium from items cut by the tips, and aluminium (+ oxygen) from the ceramic matrix (alumina – $Al_2O_3$). A variety of particles and fibres, probably surface contamination, were identified and examined. The EDX spectrum included as figure 2 clearly shows nickel, iron, chromium and aluminium peaks. A small silicon peak is clearly visible. This could be from silicon carbide fibres but could equally be from industrial contamination such as oil, cutting fluids, building dust (concrete) etc.,. There is no observable carbon peak and the oxygen peak is far weaker than would be expected. [The carbon K$\alpha$ peak at 0.3 KeV is close to the lower limit of detection of the system and is often very weak (proportional to other peaks) but the oxygen K$\alpha$ peak at 0.5 KeV is much less prone to these limitations].

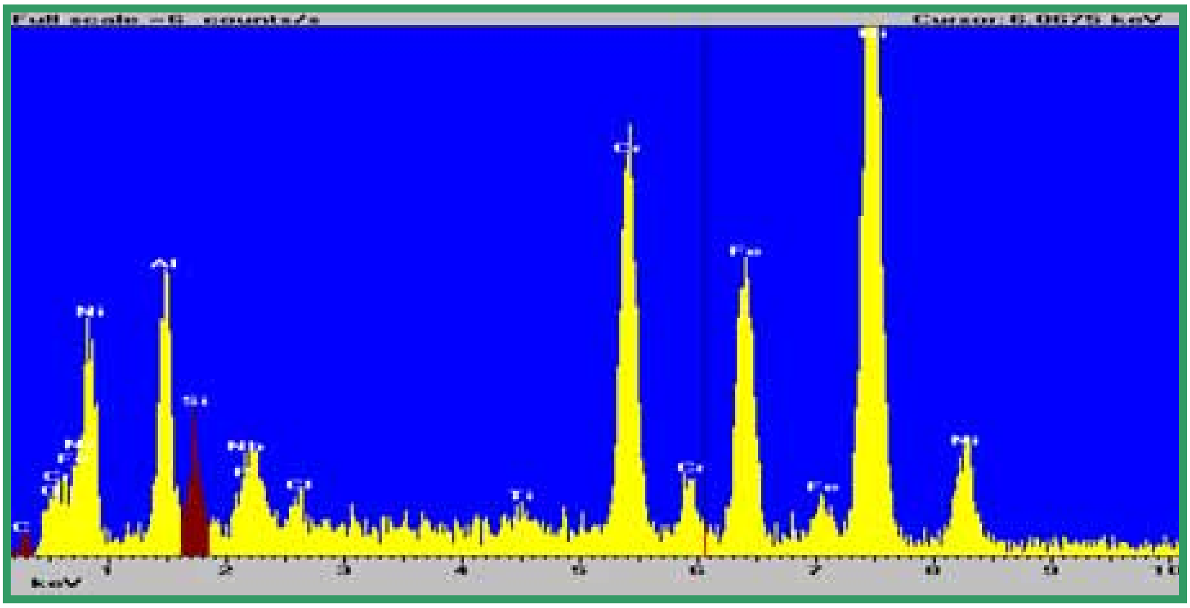


Figure 2: Typical EDX from the surface of an 'as received' silicon carbide tip.

The surfaces of the 'as received' tips, even those, which had not been used, were contaminated and finding and identifying SiC fibres on these surfaces was very difficult. The tips are extremely hard and difficult to break but at –196°C, the temperature of liquid nitrogen, the tips become brittle and it was relatively easy to produce a clean fractured surface at these temperatures. When a clean surface was produced in this way a large numbers of fine fibres (< 1µm diameter) were clearly visible (SEM). EDX spectra from these fibres indicated that the principal elemental components were silicon and carbon (SiC). The whiskers were very small, many being less than one micrometer in diameter and typically a few micrometers in length. A consequence of this small size was that the EDX spectra invariably included information from the underlying / adjacent ceramic material and aluminium and oxygen ($Al_2O_3$ - ceramic) peaks were found in all the EDX spectra. [EDX spectra from areas, which did not include fibres, did not have the silicon or carbon peaks found in spectra from the fibres].

Quantitative[5] analysis of the EDX spectra suggested that the proportions of the principal components were 31% silicon, 19% carbon, 23% aluminium and 26% oxygen. The ratio of silicon to carbon was close to that expected for silicon carbide (SiC). Similarly the ratio of aluminium to oxygen was close to that alumina (aluminium oxide - $Al_2O_3$). This analysis also

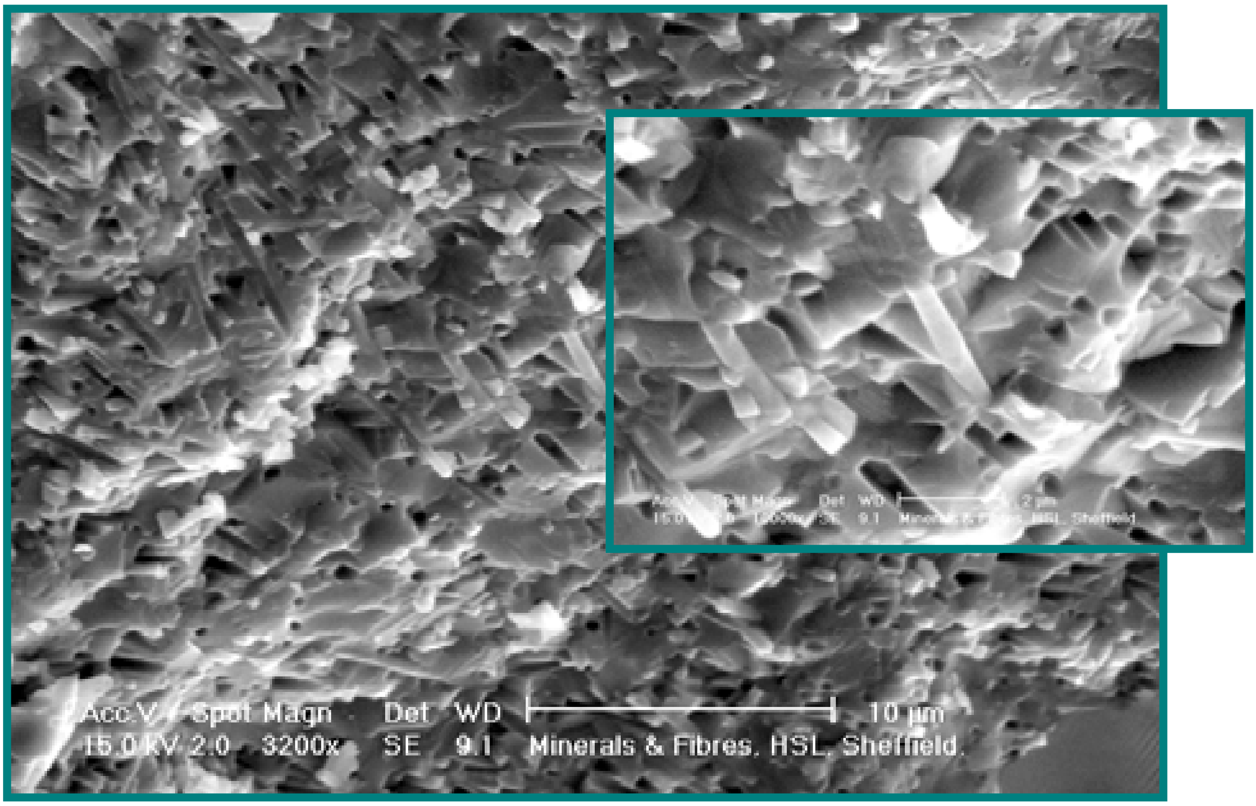


Figure 3: SiC – clean fracture surface showing SiC fibres protruding from the surface. Insert is a higher magnification image of the fibres at the centre of the main image.

[5] Oxford Instrument ISIS software can calculate elemental compositions from the EDX spectra, this quantitative data must however, be treated with caution. Only a very small area of the surface is examined but information (x-rays) is gathered from volumes greater than the dimensions of some of the finer features. Consequently the analytical results may include information from the background and/or adjacent features, in addition to the feature analysed. Carbon and oxygen peaks, which are close to the lower limit of detection for the system often, require large corrections and this quantitative data should be regarded as being for indication only.

indicated that there were approximately equal proportions of silicon carbide and aluminium oxide.

An image of the fractured surface is shown as figure 3. Large numbers of fibres, typically about 2 µm in length and about 0.5 µm in diameter, are clearly visible. These fibres are at all orientations and many appear to protrude from the fracture surface showing how the fibres are dispersed within the ceramic matrix. The release of fibres into the environment from this type of breakage would appear feasible as protruding fibres could be easily broken off. It is much less likely that similar releases would occur from a cut or worked surface, as this surface would be worn in such a manner that there would be no protruding fibres. Under normal working conditions clean fracture surfaces are extremely unlikely but Mr. Nind commented in a letter (21/06/2004) 'they (*one of the companies visited*) appeared to break tips fairly regularly'. How often this would occur and what (if any) enhanced fibre releases would result is unknown.

### 3.2 SILICON CARBIDE SIMULATION TESTS:

To simulate what happens when used in industry, a rectangular piece of the material was mounted in a drill chuck and 'rubbed' against a similar piece held in a vice. The tests were carried out inside a sealed glove box and airborne fibres released collected by sampling at 10 litre per minute for the duration of the test (2 samples). The airborne fibre concentration determined by counting the number of fibres collected using MDSH59 fibre-counting rules indicated that very few fibres were released. A total of 3 PCM fibres in 400 fields of view (area ~ 3 $mm^2$) were counted. For an air volume of 98 litre this gave a pooled airborne fibre concentration of 0.007 f/ml[6]. This was below the pooled LOQ, 0.04 f/ml, and would be reported as <0.04 f/ml, it does however, indicate that fibre releases during this type of work activity are likely to be low. Although these tests differed from the industrial use, they did simulate the cutting action. In industry the component being cut is often very hard and a great deal of heat may be generated, consequently the process is often carried out wet using water or oil based cutting oils. As well as assisting the cutting process the application of cutting oils will suppress airborne fibre releases. Consequently this test was likely to produce a 'worst case' scenario with respect to airborne fibre releases.

Table 2 – Results from Laboratory Simulation Tests.

| Simulation | Sample Nº | Air volume (litre) | Nº of fibres on filter (counted)* | Airborne fibre concentration (f/ml) | Upper 95% confidence limit |
|---|---|---|---|---|---|
| SiC | 08458/04 | 50 | 307 (1) | <0.1 (0.005) | 0.029 |
| | 08459/04 | 48 | 610 (2) | <0.1 (0.010) | 0.040 |
| | Pooled | 98 | 917 (3) | <0.04 (0.007) | 0.024 |

* the number of fibres counted in 200 fields of view (in brackets) has been extrapolated to give the total number of fibres collected over the full filter area.

The number of fibres detected was so low that the results would be reported as less than the limit of quantification. The measured airborne fibre concentrations are shown in brackets alongside the reported values.

Pooling the data from both filters increased the statistical significance (and lowers the LOQ) but the number of fibres counted was such that for the volume of air sampled the pooled airborne fibre concentration was less than the LOQ and would be reported as <LOQ.

[6] This is the pooled result from two samples of 48 and 50 litre. 1 fibre was found in 200 fields for one sample and 2 fibres in 200 fields for the other.

### 3.3 SILICON NITRIDE ($Si_3N_4$)


Figure 4: $Si_3N_4$

As an alternative to silicon carbide, silicon nitride whiskers are incorporated in some cutting tools. Some manufactures information about silicon nitride products is included in appendix 1.

No on site monitoring was carried out at factories using ceramic / silicon nitride cutting tips but a set of tips was acquired for laboratory examination. The appearance of these silicon nitride-cutting tools was very different to the silicon carbide tips. They were 13 mm x 13 mm (½ inch) square blocks, about 5 mm thick with a 4 mm central hole. Although they were gold coloured they did not have a metallic appearance. An image of a silicon nitride tip is shown as figure 4.

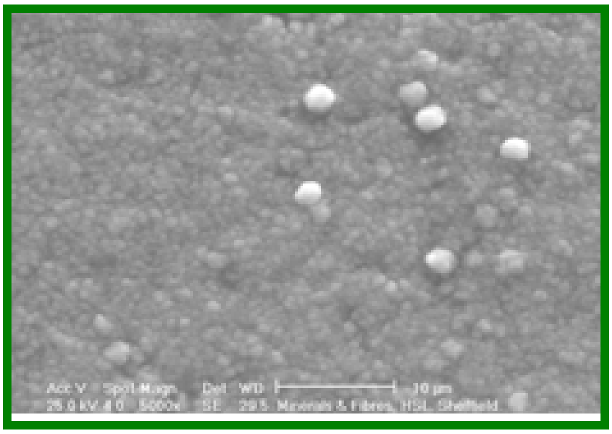


Figure 5: silicon carbide surface as received.

SEM examination of the 'as received' surface showed a marbled surface typical of some painted surface (figure 4). As with the silicon carbide tips no evidence of fibres ($Si_3N_4$) was found.

Breaking a $Si_3N_4$ tip at low temperature produced a clean fractured surface, which was light blue grey in colour. When this surface was examined using SEM techniques large numbers of fibres protruding at all angles from the surface were observed. Analysis of EDX spectra produced from these fibres indicated that they contained silicon and nitrogen ($Si_3N_4$)[7]. An SEM image clearly showing these protruding fibres is included as figure 6.

### 3.4 SILICON NITRIDE SIMULATED WORK TEST

A simulated 'work' test similar to that performed on the silicon carbide tips was carried out. In this test a tip was held in a vice and holes were drilled (or it was attempted to drill holes using a diamond tipped drill). This proved extremely difficult as the material was very hard and little progress was made. Again airborne fibres were captured on membrane filters and airborne fibre concentrations determined using MDHS 59 counting rules. The results, which are included in table 2, show that although more $Si_3N_4$ fibres were released than SiC fibres at <0.02 (0.018) f/ml the fibre release was very low.

[7] The same limitations regarding quantification apply to nitrogen as previously specified for carbon.

Table 3 – Results from Laboratory Simulation Tests For Silicon Nitride

| Simulation | Sample N° | Air volume (litre) | N° of fibres on filter (counted)* | Airborne fibre concentration (f/ml) | Upper 95% confidence limit |
|---|---|---|---|---|---|
| SiN | 04550/04 | 101 | 2,338 (7½) | <0.041 (0.018) | 0.043 |
| | 04551/04 | 101 | 2,338 (7½) | <0.041 (0.018) | 0.043 |
| | Pooled | 202 | 4,675 (15) | <0.02 (0.018) | 0.036 |

* the number of fibres counted in 200 fields of view (in brackets) has been extrapolated to give the total number of fibres collected over the full filter area.
The numbers of fibres detected were so low that the results would be reported as less than the limit of quantification. The measured airborne fibre concentrations are shown in brackets alongside the reported values.
Pooling the data from all filters (from a site) was used to increase the statistical significance but it still did not exceed the LOQ.

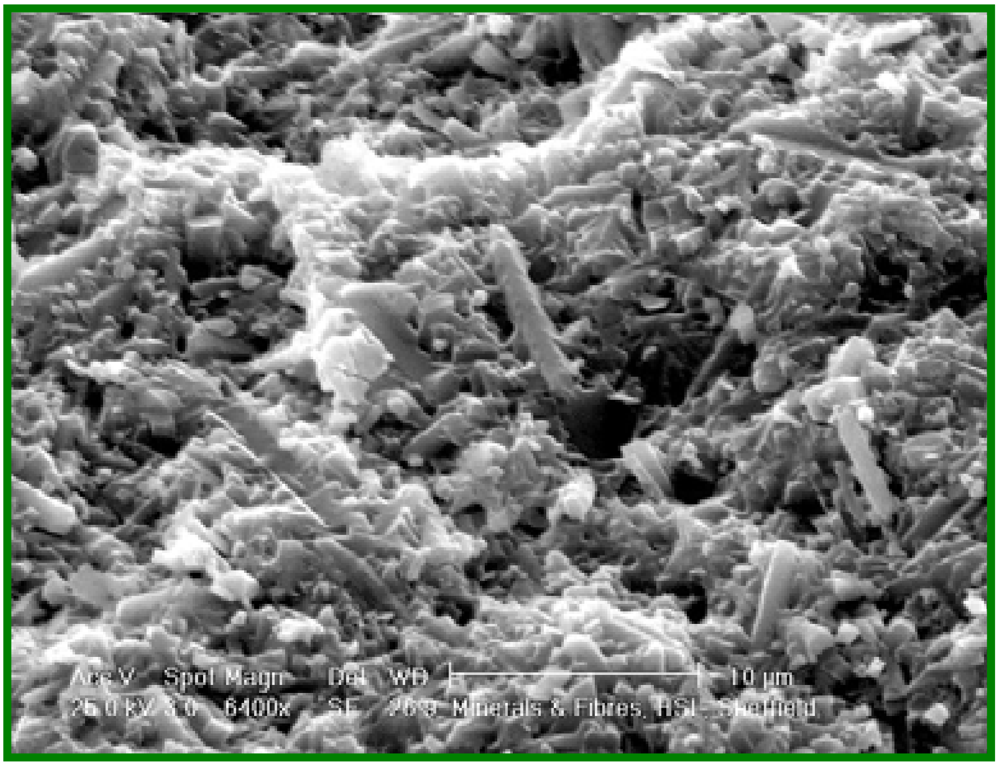


Figure 6: SEM image of silicon nitride showing fibres protruding from the fracture surface.

## 3.5 TEM EXAMINATION OF THE FIBRES.

One concern was that the airborne fibres generated in industry and in the simulation tests may be to small to be seen and counted using standard optical microscope fibre counting techniques. SEM examination of the fracture surfaces indicated that many of the fibres could be less than 0.2 µm in diameter. To determine if any of the airborne samples included very fine fibres sections of the filters were mounted for, and examined by, transmission electron microscope (TEM) techniques.

### 3.5.1 Silicon Nitride

Examination of these samples suggested that if $Si_3N_4$ fibres were present they were always associated with non-fibrous particles. It was not possible to determine if these groups of particles were loose aggregates or whether the fibres were physically attached to particulates. TEM images of typical particulate / fibre aggregates are shown as figures 7a and 7b A single detached fibre can be seen in figure 7b, this was one of the very few separate fibres found. It is presumed that the non-fibrous particulates are from the matrix within which the $Si_3N_4$ fibres were embedded.

### 3.5.2 Silicon Carbide

Very few fibres were captured in the filters either during on site sampling or during the simulation exercises carried out at HSL. An attempt to determine the airborne fibre concentration generated by the simulations was made. Eighteen graticule areas, (0.176 $mm^2$) were examined and fourteen possible, very fine (<0.5 µm diameter) SiC fibres were identified. Making various assumptions (cross section of the fibres) and approximations, this suggests that the total weight of silicon carbide fibres collected was about forty nano-grammes. Attempts to discriminate fibres by EDX techniques were made. The EDX spectrum of the fibres clearly showed a silicon (Si Kα) peak but there was no observable carbon (C Kα) peak. This is because the system is unable to detect the low energy x-rays generated from low atomic number elements (elements with atomic numbers below 12).

### 3.5.3 TEM Analysis

TEM analysis of the sample indicated that small numbers of very fine SiC or $Si_3N_4$ fibres might be present in the airborne dust generated during the simulation exercises. However, if they are present, they are only present in very small numbers and in the case of $Si_3N_4$ fibres they are usually part of an aggregate of mainly non-fibrous material. The fibres detected using TEM techniques would be too fine to be counted using the regulatory optical microscope techniques used to monitor and assess airborne releases. [These fine fibres would be assume, as for asbestos, not to present a risk to health].

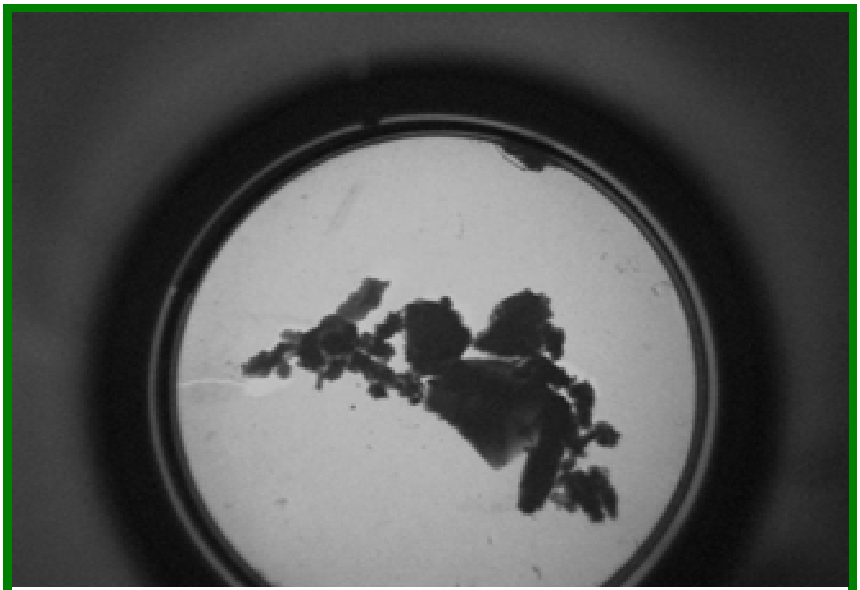
Figure 7a: TEM image of $Si_3N_4$ fibres.


Figure 7b: TEM image of $Si_3N_4$ fibres.

# 4 CONCLUSIONS

Both the silicon carbide and silicon nitride reinforced tips contain fibres, which will protrude from the surfaces if the components are fractured. It is possible that these fibres could be broken off as entities and become airborne with subsequent concerns over health. However, both industrial monitoring and laboratory simulations indicated that only low levels of airborne fibres were created during normal usage. The grinding and cutting processes for which they are used would wear away the fibres (and matrix) rather than release them as whole fibres. This is especially so when combined with dust suppression measures such as wetting and makes substantial airborne fibre releases unlikely. Breaking the tips can and does occur, and could result in enhanced fibre releases but these types of breakages are infrequent and are unlikely to result in substantial fibre release. It is unlikely that the process, as currently carried out, would pose a long term risk to health.

# 5 APPENDIX 1: MANUFACTURER INFORMATION REGARDING SILICON NITRIDE PRODUCTS.

Silicon nitride is a strong, tough, lightweight ceramic material ideal for structural applications. When processed using the proprietary techniques developed by Ceradyne, Inc., an advanced technical ceramic manufacturing company located in Costa Mesa, California, it has outstanding mechanical reliability along with high temperature, wear and corrosion resistance.

Ceradyne's silicon nitride is being used as cam rollers for valve train applications in heavy-duty diesel engines, as well as in high-pressure common rail fuel pumps and unit fuel injection systems where conventional metal parts fail. Ceradyne silicon nitride cam rollers can successfully operate at contact stresses of 1.0 to 2.4 GPa (150,000 - 350,000 psi) with no measurable wear to the silicon nitride or the companion metallic components. Ceradyne's silicon nitride is also used as rolling elements in hybrid roller and ball bearings.

The outstanding and unique combination of mechanical, chemical, electrical and physical properties of silicon nitride also makes it an ideal material for industrial wear applications. Successful applications encompass many industries and varied operating conditions such as:

- Wear surfaces in the "wet end" of paper making machines
- Check valves for "down hole" pumps for the oilfield industry
- Thermocouple tubes for the molten aluminium industry and the petrochemical industry
- Tube sheet boiler ferrules for the petrochemical industry
- Insulators for jet engine ignitors
- Positioning pins for nut welders for the automotive industry
- Cutting tools for cast iron and superalloys
- Forming and guide rolls for metal forming
- Silicon nitride replaces various metal, tungsten carbide and other ceramic materials for these applications.

Ceradyne has developed high-volume manufacturing methods that allow it to produce these types of components cost effectively and in high volume to very tight tolerances.

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# Noralide®NBD-200 Hot Pressed Silicon Nitride

## Technical Data

## Product Description

Noralide® NBD-200 Hot Pressed Silicon Nitride ( $Si_3N_4$) offers a combination of characteristics that make it an ideal material for seal face components in nuclear applications as well as other applications where maximum fracture toughness and flexural strength are required. Hot-pressed for maximum
strength and density, Noralide® $Si_3N_4$ provides better fracture resistance
than other ceramic materials. Noralide® $Si_3N_4$ meets the requirements of these Nuclear Quality Assurance Standards:

- NQA-1
- NQA-1a
- 10CFR-50, Appendix B

## Applications

Examples of Noralide® seal face components for nuclear applications include:

- Boiler water reactor (BWR) pumps
- Pressurized water reactor (PWR) pumps
- Corrosive borated water service

In addition, Noralide® $Si_3N_4$ is suitable for seal face and other components in demanding rotating equipment applications such as compressors, engines, generators, motors and turbines.

## Material Characteristics

- Good high temperature strength
- Maximum fracture toughness
- High hardness and wear resistance
- Good creep resistance
- Low density
- Little oxidation at elevated temperatures
- Low thermal expansion coefficient
- Not wetted by molten metals

Noralide® NBD-200 Hot Pressed Silicon Nitride

**Noralide® NBD-200 Hot Pressed $Si_3N_4$ Typical Physical Properties**

Saint-Gobain Ceramics application engineers can assist you with the design of cost-effective highperformance components for your specific need.

The information, recommendations and opinions set forth herein are offered solely for your consideration, inquiry, and verification and are not, in part or total, to be construed as constituting a warranty or representation for which we assume legal responsibility. Nothing contained herein is to be interpreted as authorization to practice a patented invention without a license. Noralide® is a registered trademark of Saint-Gobain Ceramics


| | |
|---|---|
| Saint-Gobain Ceramics<br>Structural Ceramics<br>Hexoloy® Products<br>23 Acheson Drive<br>Niagara Falls, New York 14303 | Telephone: 716-278-6233<br>Fax: 716-278-2373<br>E-mail: scd.sales@saint-gobain.com<br>www.hexoloy.com |




| Property | Units | Value |
|---|---|---|
| **Composition*** | | **Si•N** |
| **Grain Size** | **µm** | **<2** |
| **Density** | **g/cm$^3$** | **3.18** |
| **Hardness (Vickers)**** | **GPa** | **16** |
| **Flexural Strength 4 pt @ RT***** | **MPa** | **800** |

| | | |
|---|---|---|
| **Compressive Strength @ RT** | **MPa** | **3500** |
| **Modulus of Elasticity @ RT** | **GPa** | **320** |
| **Color** | | **Gray** |
| **Coefficient of Thermal Expansion RT to 1000°C** | **$X10^{-6}$ mm/mmK** | **2.9** |
| **Thermal Conductivity @100°C** | W/mK | 29 |
| **Electrical Resistivity @ RT*** ** | ohm-cm | $>10^{12}$ |
| **Purity** | | % |
| **$Si_3N_4$** | | 95 |
| **Al** | | 0.35 |
| **C** | | 0.50 |
| **Ca** | | 0.01 |
| **Fe** | | 0.02 |
| **Mg** | | 0.6 |
| **O** | | 2.5 |
| **Other trace elements** | | trace |
| ***Composition code:** | **Si = free silicon metal;<br>N = nitrogen;<br>Si3N4 = silicon nitride** | |
| ****Vickers 10 kg load** | | |
| ***Test Bar Size: 3 x 4 x 45 mm (0.118"x 0.157"x 1.772") | | |

# 6 APPENDIX 2: SILICON CARBIDE MANUFACTURES INFORMATION.