SRBSN MATERIAL DEVELOPMENT FOR AUTOMOTIVE APPLICATIONS

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SRBSN MATERIAL DEVELOPMENT FOR AUTOMOTIVE APPLICATIONS

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ABSTRACT

The development of a gas pressure sintered reaction bonded silicon nitride (SRBSN) manufacturing process capable of producing near net shape blanks for automotive applications is discussed.

The effects of grinding parameters on material strength, rolling contact fatigue life and friction were determined. Based on this, cost-effective grinding and finishing techniques required to finish components to automotive application tolerances were developed.

Examples of high volume SRBSN automotive components are presented. Components operating at high Hertzian contact stresses for extended periods, where metal components fail, have been proven as successful automotive applications for SRBSN ceramics.

INTRODUCTION

Silicon nitride has been identified since the 1970’s as a material that would find wide application for various engine components due to its high temperature mechanical properties, thermal shock resistance, tribological and wear properties. Katz1 summarizes a number of engine components that did go into high volume production including glow plugs, turbocharger rotors and cam roller followers. However, the number of production components is small when compared to the number of prototypes that have been successfully evaluated. Cost and reliability are the principal barriers to introduction of silicon nitride components in high volume automotive applications relative to components made from metal or other advanced materials.

The principal factors affecting the cost of silicon nitride components are the raw powder and component finishing costs. Efforts to reduce cost often lead to compromises that adversely affect the component reliability.

This paper will describe the development of sintered reaction bonded silicon nitride (SRBSN) that has successfully addressed the cost barriers and has been successful in high volume manufacturing of finished high performance components.

SINTERED REACTION BONDED SILICON NITRIDE (SRBSN)

SRBSN was developed on an R&D scale at the Ford Motor Company2,3 in the late 1970’s, and was scaled up for production by Ceradyne, Inc. in the late 1980’s. Figure 1 compares the SRBSN to the conventional sintered silicon nitride (SSN) process.

<table>
<thead>
<tr>
<th>SRBSN</th>
<th>SSN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw Materials</td>
<td>Raw Materials</td>
</tr>
<tr>
<td>(Silicon + Additives)</td>
<td>(Silicon Nitride + Additives)</td>
</tr>
<tr>
<td>Spray Drying</td>
<td>Spray Drying</td>
</tr>
<tr>
<td>Fabrication</td>
<td>Fabrication</td>
</tr>
<tr>
<td>Nitriding</td>
<td></td>
</tr>
<tr>
<td>(3Si + 2N₂ → Si₃N₄)</td>
<td></td>
</tr>
<tr>
<td>Gas Pressure Sintering</td>
<td>Sintering</td>
</tr>
<tr>
<td>10.3 MPa (1500 psi)</td>
<td></td>
</tr>
<tr>
<td>N₂ pressure</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1. Comparison of the SRBSN and SSN Processes.

The SRBSN process begins with the use of an inexpensive raw material - silicon powder - providing a significant cost advantage: silicon costs are far less than $10/kg, compared to over $80/kg for Si₃N₄.

The SRBSN process also exhibits a significantly lower shrinkage (10-12% versus 17-21%) and better dimension control than the SSN process. This results in a reduced grind stock, and consequently lower machining costs. Lower component shrinkage also allows more efficient use of high temperature furnace space relative to the SSN processing route, providing an additional cost advantage.
The gas pressure sintering process results in a material having a density of >99.3 % of theoretical, along with a microstructure composed of interlocking needles (Figure 2). This structure is responsible for the excellent fracture toughness (5.5 – 7.0 MPa m$^{1/2}$) exhibited by SRBSN.

Use of neural networks were employed to optimize numerous sintering parameters; the result being a material with a robust sintering window (Figure 3). This wide process window results in high process yields. The result of this SRBSN development is a material that exhibits the properties summarized in Table 1.

**Machining of SRBSN**

Machining studies, using design of experiment (DOE) techniques, have been conducted on Ceradyne SRBSN materials. Effects of machining direction, diamond wheel grit size, table speed, and material down-feed and in-feed per pass on the strength of rectangular ASTM C1161 size B MOR (Modulus Of Rupture) bars were initially investigated. In 1997, evaluation of effects of grinding conditions on cylindrical MOR bar specimens were started, including the design of the bar geometry and its fixture. The objectives of these studies were to increase material removal rates (decrease machining costs) and optimize the material performance.

It was found that strengths of bars machined longitudinally were not significantly affected by grinding conditions.

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**Table 1. Typical Properties of Ceradyne's Needlelok™ SRBSN**

<table>
<thead>
<tr>
<th>Grade</th>
<th>Ceralloy® 147-31N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>3.21 g/cm$^3$</td>
</tr>
<tr>
<td>Characteristic Strength</td>
<td>750-830 MPa</td>
</tr>
<tr>
<td>Weibull Modulus</td>
<td>15-25</td>
</tr>
<tr>
<td>Hardness (HV5)</td>
<td>1500 kg/mm$^2$</td>
</tr>
<tr>
<td>Fracture Toughness</td>
<td>5.5 – 6.5 MPa m$^{1/2}$</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td>25 W/m K</td>
</tr>
</tbody>
</table>

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**Figure 2. Optical microstructure of 147-31N a. polished, b. etched sample.**

**Figure 3. Neural Network Sintering Response Surfaces for Density and Strength.**

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Machining damage was significant for ASTM C1161 Size B MOR bars with a transverse machining direction. Diamond wheel grit size was the dominant factor for transversely ground bars, reducing the material strength by an average 56 Mpa when an 80 grit diamond wheel instead of a 120 grit was used (Figure 4). Figure 4 also shows a strong interaction between table speed and down feed, indicating that higher removal rates (table speed x down feed) actually improve the strength.

Wheel grit also affected the transverse ground strength of cylindrical bend specimens (Figure 5). In this figure, longitudinally ground specimens were compared to transversely ground ones (600 and 320 grit diamond)\(^5\).

Fracture origins of the transverse ground bars confirmed that subsurface damage (10-80 \(\mu\)m sharp cracks) was the predominant failure mode\(^5\).

Based on this data, and the component application, grinding conditions and sequences can be generated to optimize the component performance and minimize the machining costs.

In cases where the component strength is critical, post-machining treatments have been developed which restore the inherent material strength (Figure 6).

**ROLLING CONTACT FATIGUE**

The rolling contact fatigue (RCF) behavior of Ceralloy\(^1\)
147-31N silicon nitride has been studied using a ball-on-rod RCF machine in addition to accelerated component rig testing. The results, summarized in Table 2, show that no failures were observed up to 6.07 GPa (880 Ksi) and 51.6 million cycles. Additional testing at higher stress levels is being conducted.

### Table 2 RCF Data for Ceralloy\(^1\) 147-31N Silicon Nitride

<table>
<thead>
<tr>
<th>Test type</th>
<th>Stress (GPa)</th>
<th>Cycles (test time)</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rig test of production component</td>
<td>1.1</td>
<td>2.45x10(^7) (3000 hrs)</td>
<td>Suspension, no failure</td>
</tr>
<tr>
<td></td>
<td>1.6</td>
<td>6.36x10(^7) (2000 hrs)</td>
<td>&quot;</td>
</tr>
<tr>
<td></td>
<td>1.5-2.4</td>
<td>6.3x10(^7) (1000 hrs)</td>
<td>&quot;</td>
</tr>
<tr>
<td>RCF machine</td>
<td>6.07</td>
<td>5.2 x10(^7) (100 hrs)</td>
<td>&quot;</td>
</tr>
</tbody>
</table>

**Figure 4. Design of Experiments Major Effects and Interactions on the SRBSN Strength of Transversely Ground ASTM C1161 MOR Bars.**

**Figure 5. Effects of diamond grit and orientation on 147-31N strength – cylindrical bend specimens\(^5\).**

**Figure 6. Post machining treatment effects on transversely ground 147-31N ASTM C1161 (B) bars. A 320 grit wheel was used on all bars.**
FRICITION COEFFICIENTS AGAINST STEEL

Friction torque of Ceradyne Ceralloy® 147-31N tappet inserts and two other sintered silicon nitride materials were measured against a steel cam-lobe. Engine oil without a friction modifier was used as a lubricant. Tappets were initially diamond super-finished. Some parts were then finished by two techniques (chemo-mechanical-CMP and “Ford” proprietary finish). Friction results showed that, of the materials tested, only 147-31N Si₃N₄ offered significantly reduced friction against the cam lobe at all RPM values tested and for all finishes – Figure 7a. Sintered Si₃N₄ (SSN) grades showed a friction coefficient reduction for the “Ford” proprietary finishing technique only, which was applied as an additional manufacturing step after diamond super-finishing (Figure 7b).

The differences in friction between SRBSN and SSN materials and steel cannot be explained by their surface finishes, and are not completely understood. It is believed that the material composition and microstructural differences contribute to the different tribological behavior.

The use of Si₃N₄ tappet inserts instead of steel could offer 0.5% fuel economy benefits, in addition to a wear reduction in the valve train.

COMPONENTS

Ceradyne has been producing silicon nitride ceramics for industrial applications since 1990 and has produced over half a million parts annually for wear applications from 1996 through 1999.

Ceradyne has participated in a number of ceramic automotive component qualification programs in the U.S. since the early 90’s, with the various prototype components illustrated in Figure 8. These programs include:

- Silicon nitride exhaust valves and clevis pins for heavy-duty diesel applications. The culminated in a successful NATO durability qualification test for the engine.

- Silicon nitride tappet inserts for piston applications. Test results have shown that the use of silicon nitride inserts results in reduced friction relative to the standard metal inserts (Figure 7 and 8).

Figure 7. Friction between steel cam lobe and steel or Si₃N₄ tappet insert. a. for 147-31N Si₃N₄; b. for a European grade of sintered Si₃N₄ - redrawn from 8.
Ceradyne has been successful in qualifying its silicon nitride for high volume automotive production applications. These include:

- Silicon nitride **flat and radiused valve lifters** for racing applications, Figure 9a. The customer is selling these parts commercially for high performance after-market applications in both drag and stock car racing.

- Silicon nitride **cam rollers** for heavy-duty diesel engines, Figure 9b. These components have been tested at contact stresses of 2,400 Mpa for time periods exceeding 1000 hours.

- Silicon nitride **rolling elements** for fuel pumps in light duty diesel engines, Figure 9b. These components have been tested at contact stress levels of 1100-1600 MPa.

These examples demonstrate that Ceradyne’s Si₃N₄ material has the capability of routinely withstanding high Hertzian stresses. This is a result of a virtually pore free microstructure of its Si₃N₄, achieved by gas-pressure sintering (Figure 2a). This combined with Ceradyne silicon nitride’s high fracture toughness, results in a reliable material for high performance applications.

**CONCLUSIONS**

New emissions requirements for heavy-duty trucks will require engines to operate at higher fuel injection and cylinder pressures¹. This, combined with the trend for longer warranty periods, often extending to 1 million miles, will open up new opportunities for ceramic components in the wear and tribology areas. Ceradyne SRBSN is a proven, cost-effective material for the above demanding engine operating conditions.

Integration of cost-effective SRBSN manufacturing with high production volume machining capabilities, and experience in proven engine component applications, make Ceradyne a leader in the field of high performance automotive ceramics.
References

4. NIST Ceramic Machining Consortium, Appendix to the 10th Program Review Meeting, Gaithersburg, MA, April 10-11, 1997
5. G. Quinn; L. Ives; P. Koshy; “Cylindrical Rod Flexure Test Method and Results,” NIST Ceramic Machining Consortium, 14th Program Review Meeting, Worthington, OH, April 15-16, 1999