F. Life Prediction of Diesel Engine Components

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Objective

- Generate database and characterize damage mechanisms of candidate advanced ceramics and intermetallic alloys.
- Apply and verify probabilistic life prediction and components design and verification for advanced diesel engine components.

Approach

- Evaluate the dynamic fatigue and rotary bending fatigue performance of candidate silicon nitride ceramics and titanium aluminide (TiAl) alloys at elevated temperatures in air before and after long-term exposure to simulated engine environments, as well as engine field tests.
- Characterize the evolution and role of damage mechanisms, and changes in microstructure linked to the long-term mechanical performance and reliability of ceramics and intermetallic alloys.
- Predict the failure probability and reliability of complex-shaped components subjected to application conditions via the use of life prediction codes.

Accomplishments

- Completed mechanical properties of prototype NT551 silicon nitride exhaust valves after 500-h bench rig test
- Completed dynamic fatigue study for Kyocera SN235P silicon nitride, fabricated from the same powder batch for exhaust valve blanks, after 100-h oil immersion test.
- Completed development of dynamic fatigue database for a commercial-grade silicon nitride, SN147-31E, acquired from Ceradyne, Inc
- Completed mechanical property verification for flexure test bend bars machined from Kyocera SN235 exhaust valve blanks for mechanical performance verification.

Future Direction

- Characterize microstructure and mechanical properties of Kyocera SN235P silicon nitride exhaust valves after engine field demonstration at the National Transportation Research Center (NTRC) and verify the probabilistic component design and life prediction.
- Develop a dynamic fatigue database for Ceradyne SN147-31N silicon nitride fabricated from the same power batch for exhaust valve blanks.
- Verify mechanical properties for Ceradyne SN147-31N silicon nitride machined from the exhaust valve blanks.
- Develop the mechanical database for the specimens extracted from diesel particulate filter (DPF) substrates for long-term mechanical reliability and life prediction.

Introduction

There has been considerable interest in the potential for extensive use of advanced ceramics and intermetallic alloys in advanced diesel engine systems because of their superior thermomechanical properties at elevated temperatures. The implementation of components fabricated from these advanced materials would lead to significant improvement in engine efficiency, long-term durability, and reduction in nitrogen oxides and CO exhaust emission as required in the 21st Century Truck Program. This interest has focused primarily on research aimed at characterization and design methodology development (life prediction) for advanced silicon nitride ceramics and TiAl alloys in order to manufacture consistent and reliable complex-shaped components for diesel engine applications. The valid prediction of mechanical reliability and service life is a prerequisite for the successful implementation of these advanced materials as internal combustion engine components.

This research project has three primary goals, which contribute toward successful implementation:

- the generation of a mechanical engineering database from ambient to high temperatures of candidate advanced materials before and after exposure to simulated engine environments
- the microstructural characterization of failure phenomena in these advanced materials and components fabricated from them
- the application and verification of probabilistic life prediction methods using diesel engine components as test cases

For all three stages, results will be provided to both the material suppliers and component end-users to refine and optimize the processing parameters to achieve consistent mechanical reliability, and to validate the probabilistic design and life prediction of engine components made from these advanced materials.

Approach

All silicon nitride test bend bars were longitudinally or transversely machined according to ASTM standard C1161 from production billets purchased from material suppliers.[1] Flexure testing was conducted in ambient air in four-point bending using 20/40- mm, α-SiC, semi-articulating fixtures at temperatures ranging from 20 to 1000°C and at a stressing rate of 30 MPa/s and 0.003 MPa/s. The 30 MPa/s test condition was chosen to evaluate the inert characteristic strength as a function of temperature; the 0.003 MPa/s test condition was chosen to measure the change in slow crack growth (SCG) susceptibility at elevated temperatures. Pneumatic actuators were programmed to produce the desired loading rate (and corresponding stressing rate) via a personal computer. Load was continuously measured as a function of time, and flexure strength was calculated using ASTM standard C1161. The accumulated strength data were then further analyzed. The strengths for each test set were fit to a two-parameter Weibull distribution using the program CERAMIC,[2] which uses the maximum likelihood estimation advocated in ASTM C1239.[3] Reported results are uncensored because fractography analysis was not conducted in detail to identify strength-limiting flaws for all of the bend bars tested. Following the dynamic fatigue test, both optical and scanning electron microscopy analysis were carried out on fracture surfaces and polished cross-sections of selected bend bars to characterize the fracture as well as degradation mechanisms. X-ray analysis was also carried out to evaluate the possible phase changes resulting from oxidation during dynamic fatigue testing or after long-term exposure to simulated engine environments, which could possibly cause degradation in mechanical performance and reliability.

Results

Dynamic Fatigue Response of Kyocera SN235P Silicon Nitride

Kyocera SN235P silicon nitride was down-selected by Caterpillar for engine demonstration because of its excellent resistance to slow crack growth and to a simulated diesel engine environment at elevated temperatures. To ensure a consistent microstructure and chemistry in the secondary phase, and thus mechanical reliability, of the valve blank materials, bend bars were machined from co-processed billets for long-term oil immersion as well as exhaust gas study. Bend bars were also machined from the valve blank itself for mechanical comparison. The results obtained would also allow Caterpillar to verify its probabilistic component design and life prediction tasks. The test silicon nitride samples were placed in platinum crucibles and covered with commercial-grade 10W30 engine oil. The crucibles with oil-covered silicon nitride bend bars were heated in a furnace at 600°C for approximately 30 minutes to ash the oil. After completion of oil-ash conversion, the samples covered with 1–2 mm oil ash powder were then heat-treated at 850°C for 1000 h in ambient air. In addition, SN235P bend bars were exposed to exhaust gas for 1000 h. The results previously generated for the as-machined SN235P samples, co-processed with the exhaust valve blanks, showed a very high fatigue exponent (N ~ 93) at 850°C, indicative of no susceptibility to the SCG process.[4,5] Therefore, the dynamic fatigue tests for exposed SN235P samples were carried out only at 850°C and 0.003 MPa/s in air according to ASTM C1465 because of the limited number of specimens.

Results of tests carried out at 20 and 850°C and at 30 MPa/s showed that the SN235P valve blank material exhibited very consistent mechanical characteristic strength and Weibull modulus with respect to those SN235P materials evaluated previously in the program. Dynamic fatigue tests at 850°C and 0.003 MPa/s show that both of the oil-immersed as well as exhaust-gas exposed SN235P silicon nitride specimens exhibit an inert characteristic strength comparable to those obtained from the as-machined samples tested under the same condition (Table 1). Also, results show that there is no difference in characteristic strength values between oil-ash–exposed and exhaust-gas–exposed samples. In addition, both of the exposed SN235P samples showed a higher Weibull modulus than the as-machined samples. The higher Weibull modulus of exposed samples could arise from the surface sealing effect due to the formation of oxide scale after exposure. The dynamic fatigue results suggest that the SN235P-CP exhibits excellent corrosion resistance to diesel engine environments and no susceptibility to the SCG process at test temperatures. Thus the SN235P material would meet the application criteria for exhaust valve components of advanced diesel engines.

Dynamic Fatigue Response of Ceradyne SN147-E Silicon Nitride

Studies of dynamic fatigue properties of a commercial-grade silicon nitride, i.e., SN147-31E, manufactured by Ceradyne Advanced Ceramic, Inc., were completed this year. The objective of this study is to extend the database generation efforts to other potential candidate silicon nitride ceramics, especially those manufactured by domestic material suppliers. The SN147-31E was processed with sintering additives (i.e., Al_2O_3 and Y_2O_3) similar to those employed for SN147-31N that were evaluated and reported previously. However, the SN147-31E contains a crystalline secondary phase achieved by post–heat treatment. Thus it is anticipated that SN147-31E would exhibit a higher temperature mechanical reliability than SN147-31N because of the presence of the crystalline secondary phase.

Dynamic fatigue results showed that the longitudinally machined SN147-13E exhibited a characteristic strength that is 20% lower than that obtained from the SN147-31N machined and tested under the same conditions (Table 2). The lower strength of SN147-31E could be due to the change in internal residual stress resulting from the crystallization process of the secondary phase(s) by post–heat treatment. In addition, the transversely machined SN147-31E showed a minor decrease (~8%) in characteristic strength as compared with the SN147-31N. The minor difference in strength between SN147-31E and –31N, which were transversely

Table 1. Summary of uncensored Weibull and strength distributions for Kyocera SN235P silicon nitride specimens longitudinally machined per ASTM C1161from co-processed billets and exhaust valve blanks before and after exposure to simulated diesel engine environments. Data generated for SN235P evaluated previously in the program are used for reference.

Material	# of spmns. tested	Stressing rate (MPa/s)	Temp. (°C)	Uncens. weibull modulus	± 95% Uncens. weibull modulus	Uncens. chrctstic strength (MPa)	± 95% Uncens chrctstic strength (MPa)
SN235P	10	30	20	32.6	18.8 49.6	792	773, 810
SN235P	10	30	850	30.3	16.5, 48.1	673	643, 691
SN235P	10	0.003	850	23.6	12.7, 38.8	641	662, 641
SN235P-CPB	30	30	20	11.94	8.58. 15.94	820	794, 847
SN235P-CPB	30	0.003	20	14.56	10.93, 18.64	741	721, 761
SN235P-CPB	30	30	850	20.56	15.16, 26.87	684	671, 697
SN235P-CPB	30	0.003	850	19.44	14.49, 25.01	621	608, 633
SN235P-EVB	15	30	20	17.58	11.39, 25.07	895	866, 924
SN235P-EVB	15	30	850	10.42	6.64, 15.23	732	691, 772
SN235P-CPB-Oil	15	0.003	850	30.40	19.47, 44.19	660	647, 672
SN235P-CPB-ExhGas	12	0.003	850	25.07	15.55, 36.81	677	659, 694

CPB: specimens were machined from co-processed billets of exhaust valve blanks.
EVB: specimens were machined from exhaust valve blanks.

machined, could be due to the similarly dominant strength-limiting flaws, i.e., surface machining flaws. However, SN147-31E exhibited a lower Weibull modulus than that obtained for SN147-31N. On the other hand, results at 20°C and 0.003 MPa/s showed that both longitudinally and transversely machined SN147-31E exhibited little or no decrease in characteristic strength as compared with results obtained at 30 MPa/s, as shown in Table 2. Results also show that the Weibull moduli were not sensitive to the stressing rate. In addition, both materials exhibited high fatigue exponents at room temperature (Figures 1 and 2), indicative of no susceptibility to the SCG process. However, results at 850°C showed that the SN147-31E exhibited a relative low fatigue exponent of 56 with respect to SN147-31N (N ~ 131), indicative of a susceptibility to the SCG process at test temperatures (Figure 3). The low fatigue exponent obtained for SN147-31E suggests an incomplete crystallization of the glassy phase during post–heat treatment. The presence of these residual glassy phase(s) could play an important role in the high-temperature mechanical performance and susceptibility to SCG.

The study of dynamic fatigue behavior in four-point bending for transversely machined SN147-31N silicon nitride after 1000 h of exposure to an oil ash environment was also completed during this reporting period. The current study was carried out to understand the effect of long-term exposure to a simulated diesel engine environment on the mechanical performance of SN147-31N. As a result of the limited number of bend bars, the dynamic fatigue tests for exposed SN147-31N samples were carried out only at 850°C and 0.003 MPa/s in air according to ASTM C1465. Results of dynamic

Table 2. Summary of uncensored Weibull and strength distributions for Ceradyne SN147-31E silicon nitride specimens transversely machined per ASTM C1161. Data of SN147-31N machined longitudinally as well as transversely are used for reference.

Material	# of spmns. tested	Stressing rate (MPa/s)	Temp. (°C)	Uncens. Weibull modulus	± 95% Uncens. Weibull modulus	Uncens. chrctstic strength (MPa)	± 95% Uncens chrctstic strength (MPa)
SN147-31N-Long	15	30	20	21.73	14.07. 31.09	836	814, 858
SN147-31N-Long	15	30	850	20.35	13.58, 28.20	777	755, 799
SN147-31N-Long	15	0.003	850	16.19	10.57, 23.02	732	706, 757
SN147-31N-Trans	15	30	20	13.76	8.96, 19.59	677	649, 705
SN147-31N-Trans	15	30	850	18.26	11.67, 26.47	639	619, 659
SN147-31N-Trans	15	0.003	850	19.95	12.83, 28.63	620	602, 638
SN147-31E-Long	15	30	20	17.30	10.86, 25.58	668	645, 690
SN147-31E-Long	15	0.003	20	16.19	10.60, 22.98	620	598, 642
SN147-31E-Long	15	30	850	9.36	6.01, 13.62	604	567, 641
SN147-31E-Long	15	0.003	850	12.06	7.70, 17.52	509	485, 533
SN147-31E-Trans	15	30	20	7.44	4.89, 10.58	623	575, 671
SN147-31E-Trans	15	0.003	20	8.99	5.61, 13.45	607	568, 646
SN147-31N-1000hOil*	15	850	0.003	15.54	10.31, 21.65	600	578, 622

*1000h Oil: specimens were transversely machined and exposed to an oil ash environment for 1000h.

fatigue tests at 850°C and 0.003 MPa/s showed that the exposed SN147-31N silicon nitride exhibited an inert characteristic strength comparable to results obtained from the as-machined samples tested under the same condition (Table 2). Also, there is no difference in Weibull modulus between oil-ash exposed and as-machined samples. The dynamic fatigue results suggest that the SN147-31N exhibits excellent corrosion resistance in diesel engine environments, similar to those observed for SN235 and GS44 silicon nitride ceramics evaluated previously. Therefore, one could conclude that silicon nitride ceramics, in general, exhibit superior corrosion resistance in diesel engine environments compared with those metallic alloys.

Mechanical Properties of Diesel Particulate Filters

This study, part of a cooperative research and development agreement between Cummins and Oak Ridge National Laboratory, is carried out to characterize the mechanical properties of a DPF substrate and develop analytical tools for predicting long-term reliability and durability. Miniature test samples have been successfully machined from the substrates of DPFs acquired from Corning (Figure 4a) during this reporting period. The samples have nominal dimensions of 0.20 × 0.90 × 15 mm. A miniature test fixture has also been designed and machined (Figure 4b). The alumina test fixture has inner and outer

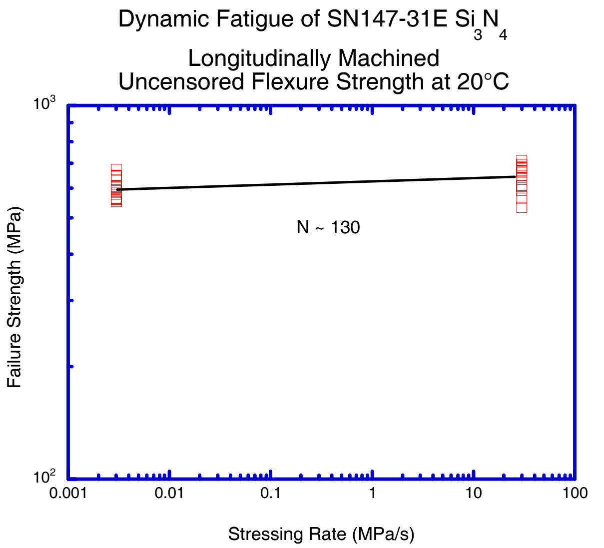

Figure 1. Failure strength vs stressing rate curve of SN147-31E longitudinally machined and tested at 20°C in air.

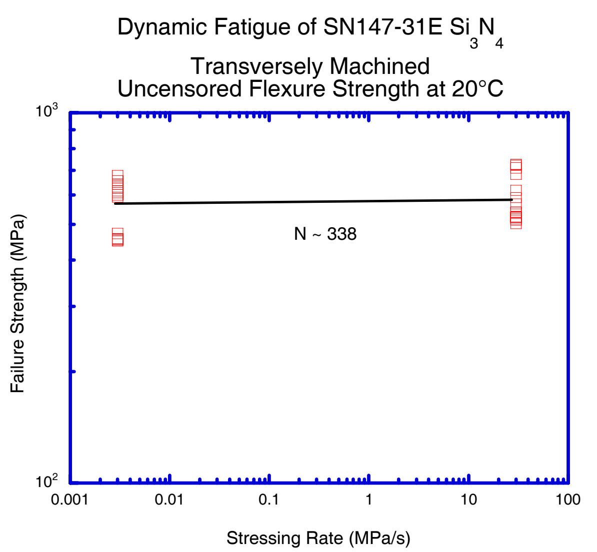

Figure 2. Failure strength vs stressing rate curve of SN147-31E transversely machined and tested at 20°C in air.

spans of 1 and 2 mm, respectively. Figure 5 shows the uncensored Weibull strength distribution of DPF samples. Results indicate that the DPF material exhibits low characteristic strength as well as Weibull modulus because of its porous microstructure. The strength data will be also generated from the full-size DPF samples for comparison. The data generated for the miniature test samples from DPF substrates would be used for life prediction.

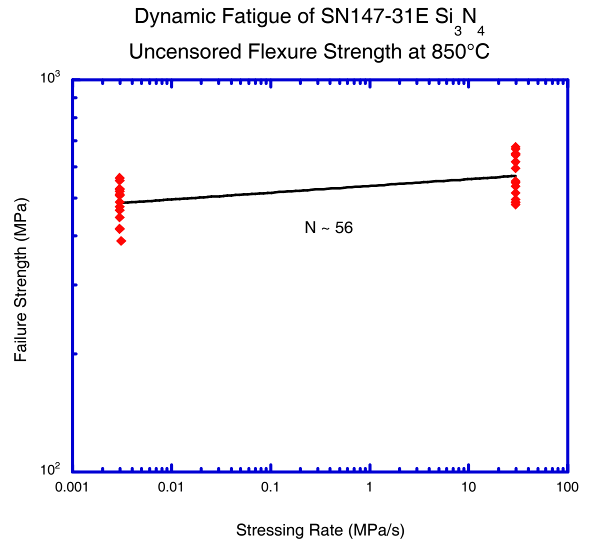

Figure 3. Failure strength vs stressing rate curve of SN147-31E longitudinally machined and tested at 850°C in air.

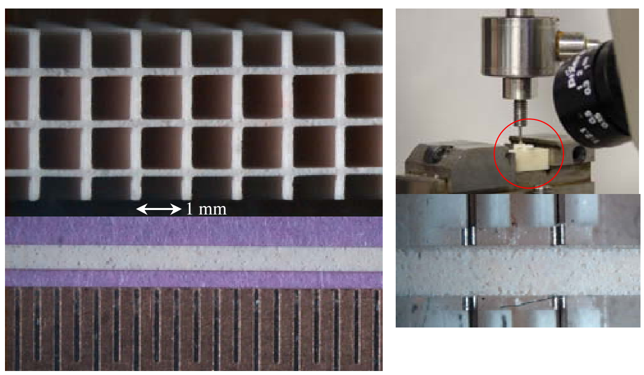

Figure 4. (a) Photos show a section of diesel particulate filter, and sample machined from it. (b) Photos show the micro test system and miniature test fixture.

References

1. American Society for Testing and Materials, "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperatures," ASTM C1161, *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing and Materials, West Conshohocken, PA, 1999.

2. *Life Prediction Methodology for Ceramic Components of Advanced Heat Engines, Phase 1*, ORNL/Sub/89-SC674/1-2, prepared by AlliedSignal

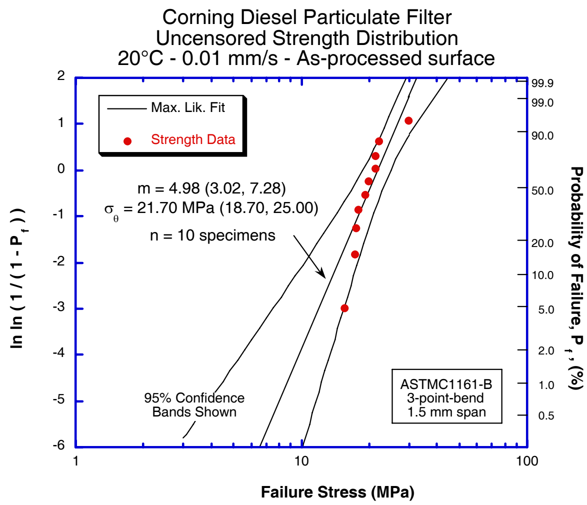

Figure 5. Failure strength vs stressing rate curve of SN147-31E longitudinally machined and tested at 20°C in air.

Engines, Department of Energy Office of Transportation Technologies, Washington, D.C., 1995.

3. American Society for Testing and Materials, "Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics," ASTM C1239, *Annual Book of ASTM Standards, Vol. 15.01*, American Society for Testing and Materials, West Conshohocken, PA, 1999.

4. A. A. Wereszczak, H. -T. Lin, T. P. Kirkland, M. J. Andrews, and S. K. Lee, "Strength and Dynamic Fatigue of Silicon Nitride at Intermediate Temperatures," *J. Mater. Sci.* **37**, 2669–2684 (2002).

5. H. -T. Lin, T. P. Kirkland, A. A. Wereszczak, and M. J. Andrews "Effect of Long-Term Oil Immersion Test on Mechanical Reliability of Candidate Silicon Nitride Ceramics for Diesel Engine Applications," pp. 261–273 in *Ceramic Transactions*, Vol. 142, "Silicon-Based Structural Ceramics for the New Millennium" (2003).

Publications

1. H. -T. Lin, T. P. Kirkland, A. A. Wereszczak, and M. J. Andrews, "Effect of Long-Term Oil Immersion Test on Mechanical Reliability of Candidate Silicon Nitride Ceramics for Diesel Engine Applications," pp. 261–273 in *Ceramic Transaction*s, Vol. 142, "Silicon-Based Structural Ceramics for the New Millennium" (2003).

2. H. T. Lin, T. P. Kirkland, A. A. Wereszczak, and M. J. Andrews, "Strength Retention of Silicon Nitride After Long-term Oil Immersion Exposure," submitted to *J. Mater. Sci.*