CFCC APPLICATIONS FOR DIESEL ENGINE VALVE GUIDES

DOE Continuous Fiber Ceramic Composite Program

Phase IIA/B

Final Report

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Executive Summary

The driving forces for diesel and alternative fuel technology are reliability, durability, efficiency, and emission regulations. The self-lubricating exhaust valve guides technology was expected to increase the reliability and durability of advanced, fuel efficient diesel engines and diesel engines which use low emission fuels. For this reason, the objective of this program was to develop a self-lubricating carbon fiber / silicon nitride composite exhaust valve guide for diesel engines. The program was divided into three phases – Phase I - Exploratory Development, Phase II - Process Engineering and Component Development, and Phase III - Pilot Scale Process Development. The results of Phase I were presented in a final report (April, 1996). This report summarizes the results of Phase II. Phase II consisted of three major tasks -- Process Optimization for Manufacturing, Component Fabrication and Testing, and Component Evaluation.

The Process Optimization for Manufacturing task focused on developing a cost-effective, production-viable valve guide manufacturing process, using the two most promising material formulations from the material screening matrix in Phase I. The formulations consist of chopped carbon fiber at two volume concentrations (10% and 12.5% Amoco P-75) in a GS-44 silicon nitride matrix. Two near-net shape forming techniques were investigated: cold isostatic pressing (CIP) and gelcasting. The CIP process is well suited for forming tubular components such as the valve guide, and has a high potential for automation. Gelcasting has several potential advantages over slip casting (used in the Phase I material screening study) including all-metal reusable tooling, shorter casting time, more uniform microstructure, and better dimensional control.

Both the CIP and gelcasting technologies had been developed for monolithic silicon nitride components; the technical challenge was to incorporate chopped fibers in uniform distribution. After a relatively short period, the CIP process produced acceptable CFCC tubes. In contrast, after the initial gelcasting experiments it became apparent that extensive development would be required to produce acceptable CFCC tubes. Therefore the gelcasting effort was eliminated, and the CIP process was refined under the Process Optimization for Manufacturing task and used to fabricate valve guides for the Component Fabrication and Testing task.

Under the Component Fabrication and Testing task, flexure test bars and prototype valve guides were fabricated for mechanical property measurements, durability testing in a simulated diesel engine test rig, and wear coefficient measurements. Mechanical property test results indicated that the CFCC material provides adequate strength for the valve guide application. The simulated diesel engine rig tests and wear coefficient measurements indicated that the CFCC and the monolithic silicon nitride guides exhibited significantly less wear than the standard cast iron guides, and the CFCC guide caused significantly less valve stem wear than either the monolithic silicon nitride guides or the standard cast iron valve guides. Based on this data, valve guides were fabricated for engine testing, to be conducted under the Component Evaluation task.

The Component Evaluation task included selection of an engine for testing the valve guide, selecting the final design for the valve guide, and testing CFCC, monolithic silicon nitride, and standard cast iron valve guides in an engine. The 3500 series engine was selected for the test, and a bimetal exhaust valve guide design was selected. After several delays, the engine test was initiated in July 1996 and had run for 224 hours when a facility problem forced a shut-down of the engine. Upon inspection it was determined that a number of the valve guides had moved from their original position during the test. Wear measurements of the guide/stem pairs indicated the CFCC performance was significantly lower than the standard cast iron valve guide/stem pairs. The engine was re-built, and the test resumed for another 600 hours. Again, measurements showed the performance of CFCC valve guide/stem pairs to be lower than standard cast iron or monolithic silicon nitride guide/stem pairs, in direct conflict with the simulated diesel engine rig test and the wear coefficient measurement tests.

A work plan to address the conflicting results was prepared by Caterpillar with input from AlliedSignal Inc., Ceramic Components and Oak Ridge National Laboratory (ORNL). Two bench tests were performed - one at Caterpillar and the other at ORNL. Both tests provided friction coefficient data versus load and lubrication conditions, and were designed to match closely the operating conditions of the 3500 series engine. After completing the ball-on-flat tests, ORNL performed surface analysis on the tested CFCC material. No protective carbon film was detected on the CFCC and ORNL concluded that the CFCC provided no performance advantage at room temperature compared to either cast iron or monolithic silicon nitride. At elevated temperature, the CFCC's performance was nearly equal to monolithic silicon nitride. At the completion of Phase II, it has been determined that the CFCC valve guide is not effectively self-lubricating. Further, it is concluded that the original goals for the CFCC Valve Guide Program will not be met. Therefore, the pilot process scale-up planned for Phase III will not be initiated.

1.0 Introduction

The objective of this program was to develop a self-lubricating carbon fiber reinforced silicon nitride exhaust valve guide for diesel engines. Carbon fiber was chosen primarily for its self-lubrication potential, and silicon nitride for its excellent mechanical and corrosion resistant properties. This program was planned originally to be conducted in three phases: Phase I – Exploratory Development, Phase II – Process Engineering and Component Development, and Phase III – Pilot Scale Process Development.

Phase I resulted in an initial processing method, slurry infiltration/blending, being selected for fabricating proof of concept test samples. A final report of Phase I activities and achievements was submitted in April 1996, concluding that the CFCC material caused less wear than monolithic silicon nitride or standard cast iron. Further development and testing was justified for Phase II.

Phase II was conducted in two sections, IIA and IIB. The Phase IIA effort focused on material property testing, developing a production-viable valve guide fabrication process, selecting an engine for valve guide testing, and completing the final design of the valve guide. The results of Phase IIA tests indicated the CFCC valve guides provided a wear advantage over valve guides made of silicon nitride or standard cast iron. In Phase IIB, the valve guide fabrication process was refined and valve guides were produced for engine testing. A machining evaluation was performed to reduce the cost of I.D. machining, the most expensive step in valve guide production. Wear properties of the CFCC valve guides were determined, and engine testing was conducted using valve guides fabricated from CFCC, silicon nitride, and standard cast iron. Engine test results contradicted the bench/lab tests that had indicated reduced wear from the CFCC material. Additional bench tests were performed to analyze the engine test results, and it was determined that the CFCC offered no advantage over silicon nitride at elevated temperature and no advantage over either silicon nitride or cast iron valve guides at room temperature.

The valve guide fabrication development results and the engine test results and analysis were expected to provide a sound basis for Phase III, Pilot Plant-Scale Up and Commercialization, but unfavorable results from engine testing preclude initiating scale-up activities. Plans to develop a pilot scale process under Phase III have been canceled.

This document is the Final Report presenting the results achieved under Phase IIA and Phase IIB.

2.0 Background

The driving forces for diesel and natural gas engine technology are reliability, durability, efficiency, and emission regulations [1]. The foundation of reliability and durability is common to both the social need for reduced emissions and the customer need for increased efficiency and reduced owning and operating costs. The exhaust valve guide was identified as a component whose reliability and durability are negatively impacted with the increased diesel engine efficiency and the use of low-emission fuels.

Engine efficiency is a primary driving force for engine technology [2]. There are existing programs to incorporate technology into engine design to increase efficiency and increase the power output. Since 1980, the usable power from an engine has increased from 36 to 43 percent. These efficiency increases translate into decreased owning and operating costs in every application using an engine as a power source.

The impact of efficiency can be shown in the power generation market where an increase of 2 percent in fuel efficiency can translate into \$30,000 to \$60,000/year savings per engine in fuel costs. Market growth is expected in power generation due to the energy needs of developing countries. Increased efficiency will help the end-user lower operating costs and when combined with low emission technologies will supply developing countries with cleaner power.

Improved fuel economy is achieved, in part, through insulation of combustion chamber components, thereby reducing heat rejection, along with the incorporation of exhaust heat recovery systems, such as turbocompound units [2]. The insulated components include piston caps, flame deck inserts and exhaust port liners. This technology also has the potential for cost effective uses in high load-factor industrial and agricultural applications where operating costs are driven by engine fuel costs.

Valve guide temperature in LHR diesel engines can exceed 650°C compared to less than 250°C in conventionally cooled engines. This is due to higher exhaust gas temperatures (>1200°C vs. 550-650°C) and higher cylinder head temperatures that result from the elimination of water cooling. Cast iron valve guides in current engines operate for 20,000 hours with commercial liquid lubricants fed into the guide-valve stem interface. At temperatures above about 300°C, however, the cast iron has insufficient yield strength to support valve stem side loads. The resultant deformation in the guide allows the valve to contact the cylinder liner and/or not seat properly in the valve seat, resulting in a decrease in the operating life and performance of the engine [3]. Another challenge for valve guides operating at temperatures above 500°C is that the available lubricants, both commercial and experimental, cannot withstand this severe thermo-oxidative environment without deposit formation and loss of friction and wear control. Excess deposits cause valve sticking and/or form into particles that accelerate abrasive wear of the valve stem and guide.

Federal emission standards enacted in 1991 and 1994 for NO_x and particulates require attainment of 5 grams/hp-hr of NO_x and 0.1 grams/hp-hr of particulate for on-

highway diesel engines. One source of particulate emissions is lubrication flowing down the valve guide and entering the combustion chamber. Incorporation of a self-lubricated valve guide would reduce the amount of lubricant in the combustion chamber which, in turn, would reduce the amount of particulate emissions.

Stricter Government environmental regulations are also causing an increase in the number of alternative fueled diesel engines. Examples of alternative fuels are methanol (M-85), propane, and compressed natural gas. These fuels are significantly cleaner burning and for this reason, will form fewer deposits during combustion. In diesel fueled engines, the combustion deposits coat a number of the critical tribological interfaces such as the valve stem and valve guide, and serve as a solid lubricant, reducing wear. In natural gas fueled engines, these deposits are not present and for this reason there is a significant amount of wear at the valve stem-valve guide interface. The incorporation of a self-lubricating CFCC guide in this engine application would reduce the amount of wear between the valve stem and valve guide because the friction coefficient between a self-lubricating guide and valve stem would be reduced. Reducing the amount of wear would increase the time between engine overhauls which, in turn, would reduce the owning and operating costs of a natural gas fueled engine.

Finally, natural gas engines are facing strong competition in Europe where competitors are increasing operating pressure and benefiting from increased efficiency [4]. In order for Caterpillar to surpass the competition and increase market share among European competitors, engine efficiency (and thus operating pressure) must rise. The higher pressure will further increase the high wear conditions for the valve stem-valve guide interface.

In view of the above, the need was identified for a self-lubricating, carbon fiber reinforced silicon nitride valve guide, which would provide both the required friction and wear properties and structural integrity under all these operating conditions.

The wear behavior of ceramic composites containing carbon is a function of the thickness and spread of the carbon lubricant film that is present between the mating surfaces. In the application under this program, the carbon film would be formed by the sliding action between the carbon containing composite surface and the mating metallic surface. The thickness of the interfacial carbon film is dependent on the carbon loading in the composite. In composites that have less than a critical amount of carbon, the sliding surface is only partially covered with a carbon film and for this reason the coefficient of friction is dependent on the composite matrix material and the mating metallic surface. On the other hand, for composites containing carbon above the critical amount, the sliding surface is completely covered with the carbon and the coefficient of friction is independent of the materials in the sliding pairs [5, 6].

The wear behavior of carbon containing composites shows that the carbon considerably reduces both wear and friction. In addition, the transition from light to severe wear occurs at significantly higher contact pressures [7]. Incorporation of a CFCC

self-lubricating valve guide would be expected to provide: 1) the required matrix strength to support the valve side loads and 2) a lubricating phase that will survive the severe thermal oxidative environment of the LHR diesel engine.

3.0 Phase IIA/B Technical Progress

3.1 Introduction

The objectives of Phase IIA/B were to determine the effect of using self-lubricating exhaust valve guides in a current production natural gas fueled diesel engine with valve guide problems, and to develop a cost effective near-net-shape forming process to fabricate valve guides. The technical efforts reported here from Phase IIA/B encompassed Task 3.2 Process Engineering, Task 3.3 Component Fabrication and Testing, and Task 3.4 Component Evaluation.

3.2 Process Optimization for Manufacturing (Task 3.2)

The objective was to establish a cost effective near net-shape forming process to fabricate CFCC valve guides. Two near-net-shape forming techniques were investigated in parallel at the start of Phase IIA: Cold isostatic pressing (CIP) and gelcasting. The two techniques were applied under Task 3.2.1 and were to be evaluated under Task 3.2.2. However, prior to the Task 3.2.2 evaluation, CIP was down-selected as the most cost-effective process to produce CFCC valve guides meeting the performance, dimensional and surface finish specifications. The CIP process for forming valve guides was optimized under Phase IIB Task 3.2.1.

3.2.1 Net Shape Fabrication Optimization

3.2.1.1 Cold Isostatic Pressing

The objective of this effort, to develop a cold isostatic pressing (CIP) technique for forming CFCC valve guides, was met under Phase IIA/B. The CIP technique was selected due to its high potential to be a low cost, high speed process to fabricate cylindrical parts, such as valve guides. Prior to this program, CIP technology had been developed at CC for the purpose of fabricating monolithic GS-44 parts, including various sized tubular parts.

The key to successful fabrication of a ceramic part by CIP is control of the feed stock powder's flow characteristics. The feed stock powder is usually prepared by a well-controlled spray drying process, such as that developed at AlliedSignal Inc., Ceramic Components for monolithic GS-44 silicon nitride. The fiber and GS-44 raw materials could not be spray dried together to prepare CFCC feed stock powder, so a blending process for the fibers and GS-44 spray dried powder was developed. To prepare for the blending operation, the fibers had to be separated into individual fibers (1/8" long) from the as-received fiber tows, without damaging the fiber integrity. Several experimental techniques were evaluated. Of these, a wet process, which involved mixing/agitation and

screening, was the first technique to achieve complete fiber separation and effective fiber length control. The wet processed fibers were dried and blended at 10 vol% loading with standard spray-dried GS-44 powder.

The effectiveness of the blending operation was evaluated by examining the microstructure of press powder feedstock. Disks (1.125 " dia.) were pressed from the CFCC mix using a uniaxial die. A microscopic examination of the as-pressed surfaces showed that the fibers were separated and uniformly distributed. Using the same blending parameters to prepare feedstock powder, two tubes (approximately 7" long x 0.75" O.D x 0.22" I.D.) were successfully formed by dry bag CIP. The pressed disks and tubes were successfully densified by hot isostatic pressing (HIP) to full density (using the thermal processing procedure established under Phase I).

However, process refinements were needed. The wet agitation process for fiber separation and sizing, while effective, was time consuming, labor intensive, and produced a low yield (approximately 70%) of fiber. A dry agitation process was developed, producing fiber separation and length reduction results better than the wet process, in less time and higher yield (approximately 90%). Batches of CFCC feed stock were prepared and CIPed, resulting in tubes with smoother and straighter O.D. surfaces. A microscopic examination of the fractured surface, which represents the interior structure, showed that fiber agglomerates were present and the fiber length was still too long.

After five iterations of processing optimization experiments from fiber separation through HIP densification, a "baseline" process was defined which produced dense tubes having uniform fiber distribution. While yield was lower than desired due to cracking during pressing and warpage during densification, the baseline process did produce final machined specimens within specifications. The process was applicable for tubes containing both 10% and 12.5% by volume of chopped carbon fiber. This baseline process was used to fabricate the test specimens and prototype valve guides for engine tests under Task 3.3 Component Fabrication and Testing. A flowchart of the valve guide fabrication process is shown in Figure 3.2.1.1-1. Views of the CFCC's isopressed and sintered microstructures are shown on the flowchart.

CFCC Valve Guide Fabrication Process

Figure 3.2.1.1-1. The baseline CFCC valve guide fabrication process is shown. A micrograph of blended material is shown on the upper right, and the microstructure of HIPed CFCC material is shown on the lower left.

Having established the baseline process, refinement efforts continued under this task in parallel with preparations for engine testing under Task 3.3. Several experiments were conducted to reduce the raw material cost by substituting lower cost fiber, but the results were poor. An alternative method of reducing material cost was to minimize the excess material needed for grind stock. This was accomplished by improving the fiber separation, batch mixing, and pressing processes, thereby reducing significantly the amount of warpage in the densified tubes. The reduction in warpage with process improvements is illustrated in Figure 3.2.1.1-2. The tube at the left shows a typical sintered tube from the baseline fabrication process. The middle tube illustrates the reduction in warpage after the improvements, and the tube on the right illustrates the reduction in grind stock (the tube was pressed in smaller diameter CIP tooling) accomplished by the improved fabrication processes.

Figure 3.2.1.1-2. Improvements to the baseline fabrication process significantly reduced the amount of grind stock required on the CFCC valve guide blank.

While warpage was significantly improved, yield remained unacceptable due to incidences of warpage and, more significantly, cracking of the tubes. These issues were addressed successfully toward the end of this task. In the meantime, the issue of process scale-up was investigated as described in the following paragraphs.

Experiments were performed to determine whether the CFCC tubes could be formed using a higher volume, production-ready technique. The wet bag CIP used in the baseline process involves filling a flexible mold (bag) with the CFCC powder mixture, immersing the bag in a fluid tank, and pressurizing the fluid to compact the powder mixture. The wet bag CIP process is desirable for small quantities of components, because tooling is inexpensive and easy to make. However, wet bag CIPing is not ideal for high volume production, as it is a labor intensive, batch process. The "dry bag" CIP technique offers the potential for automated, high volume production. Powder compaction is accomplished by air/fluid pressure at five times the rate of the wet bag process. Dry bag CIP processing requires less hands-on labor. Blanks are formed, one after the other, as long as powder is supplied to the press. Another advantage is that tubes compacted with the dry bag are uniformly dense along their entire length, resulting in less wasted material at the ends of the compacted piece.

The most significant unknown in switching to the dry bag process was its effect on the CFCC's microstructure. As an initial evaluation, existing wet bag CIP tooling was modified to fit the dry bag press and CFCC tubes were CIPed. The tubes were densified, sectioned, polished and examined under an optical microscope. There was no apparent difference in microstructure compared to wet bag CIPed CFCC tubes; the fibers were randomly oriented and uniformly dispersed. These results, from experiments using modified tooling, indicate that the CFCC tube forming process could be successfully transitioned to the dry bag process and optimized for high volume production.

Alternative thermal processing techniques also could improve production readiness and reduce processing costs. In the baseline process, tubes are densified by hot isostatic pressing (HIP). While the HIP process is highly effective, it is a batch process, relatively expensive and with no potential for automation. Gas pressure sintering was identified as a potential lower cost alternative. However, initial experiments produced poor results. CFCC tubes were gas-pressure sintered in the thermal cycles normally used to densify monolithic GS-44 silicon nitride. None of the CFCC tubes reached full sintered density in the gas pressure sintering experiments, and the carbon fibers were badly deteriorated. Further sintering experiments would be needed to determine whether a gas pressure densification cycle could be developed for the CFCC material.

As mentioned above, cracking and warpage were the chief defects causing low yields from the baseline process. In order to optimize the baseline process, an experimental matrix was designed, focusing on the first steps of the process: materials batching and blank forming. The experimental matrix, designed to isolate the effects of critical variables in those process steps.

The matrix was designed to test the effects of: 1) raw material lot-to-lot variations, 2) blended material lot-to-lot variations (GS-44 silicon nitride powder blended with 10 vol% Amoco P-75 chopped carbon fibers), 3) mold type (size of bag, core rod material, and core rod length), and 4) packing density of the powder before pressing. The results of the test matrix suggested that the incidence of cracking and warpage correlated only to factors 3 and 4 (mold type and packing density), while little or no effect was shown from factors 1 and 2 (lot variations from raw materials or blended materials).

The test matrix results indicated a 100% correlation between cracking and a gap between the core rod and the bag cap. Also, cracks occur when the core rod material is not sufficiently stiff. Both of those factors were eliminated. Consistently good results were achieved with hardened steel core rods of sufficient length so that no gap was possible between the rod and the bag cap. The primary cause of warpage was correlated to low packing density in the mold. Being controlled by the mold filling procedure, this factor was also eliminated. Sufficient packing density of the powder mix can be achieved by tapping the mold as it is filled, as demonstrated in the experimental results. Warping occurred in tubes that were not tapped while the mold was being filled, but none of the tubes warped that were tapped during filling.

In summary, a baseline process for fabricating CFCC tubes was established and optimized during the execution of this task. The primary factors were identified which control warpage of the CIPed tubes and yields from the process. The process was revised accordingly, resulting in significant improvements. The potential for automating the forming process by dry bag CIPing was indicated by preliminary pressing experiments. Further experimentation would be required to develop an effective gas pressure densification cycle that could replace the baseline glass encapsulation HIP process.

3.2.1.2 Gel Casting

The objective of this effort was to develop a near-net shape forming technique for fabrication of valve guides using gelcasting. The potential advantages of this technique over conventional slip casting, which was used in Phase I material development, include the use of more production-oriented, all-metal reusable tooling, shorter casting time, and more uniform microstructure and better dimensional control.

A baseline gelcasting technology was developed for monolithic GS-44 (under other development programs at CC) for various near-net shape components, including tubular parts. The approach for forming CFCC tubes was first to prepare the baseline monolithic GS-44 slurry with gelling agents and then blend the fibers into it to form a homogeneous composite slurry before adding the gelling initiators. However, none of the first attempts to gelcast CFCC material produced a homogeneous composite casting.

It became apparent that the CFCC slip/slurry preparation procedure for gelcasting process would need significant development work to produce a uniform distribution of

the carbon fiber. Meanwhile, very good results from the CIP process effort were achieved. The work on developing a gelcast CFCC material was therefore discontinued.

3.2.2 Evaluation

This effort was to evaluate CFCC specimens fabricated under Task 3.2.1 Near-Net-Shape Fabrication Optimization using either dry bag cold isostatic pressing (CIP) or gelcasting. Wear coefficients were to be generated using the Cameron-Plint reciprocating friction and wear test machine. The operating conditions for this friction and wear testing (temperature, loads, and lubrication) were to be determined by Caterpillar. The wear specimen geometries were to be an AISI 4140 valve stem running against a quarter section of the silicon nitride – carbon fiber composite exhaust valve guide. Finally, the data and processing were going to be analyzed and used to select between the two fabrication processes for further optimization under Phase IIB. The CIP process was selected so no process down-selection based on friction coefficient was needed.

3.3 Component Fabrication and Testing (Task 3.3)

3.3.1 Specimen and Prototype Valve Guide Fabrication

3.3.1.1 Property Specimens

The objective of this effort was to fabricate CFCC plates (at the two selected fiber loadings), using the Phase I process (pressure slip casting), for machining into test bars for characterization under Task 3.3.3 Mechanical Property. The required number of plates (2" x 3" size) were fabricated through HIP densification at CC, and shipped to Caterpillar for machining and characterization.

However, due to the very good CIP results discussed in Task 3.2.1, it was decided not to proceed with machining and characterization of the slip cast specimens. To better represent the properties of the prototype valve guides, the test specimens were machined from CIPed tubes. Two tubes of 10% by volume P-75 fiber in GS-44, two tubes of 12.5% by volume P-75 fiber in GS-44, and one tube of monolithic GS-44 were shipped to Caterpillar for machining and testing, which was conducted under Task 3.3.3.

3.3.1.2 Prototype Valve Guides

The original work plan for this task was to use the process developed under Phase I (pressure slip casting) to fabricate prototype valve guides for durability testing under Task 3.3.4 in a simulated diesel engine test rig. For comparison purposes, the test plan included valve guides of baseline cast iron, monolithic GS-44, and the two CFCC compositions selected under Phase I. Two large CFCC plates (approximately 5" x 5" x 1.2") were slip cast, from which tubes would have been machined. However, the work plan was revised following the promising results from Task 3.2.1. With approval from the DOE Program Manager, the fabrication method was changed to CIP. The prototype CFCC valve guides were machined instead from tubes fabricated by the baseline process described in Task 3.2.1.

For bench testing, twelve tubes, four with no carbon fiber, four with 10% by volume of carbon fiber, and four with 12.5% by volume of carbon fiber, were CIPed, partially densified (presintered), presinter machined, fully densified (sintered in the hot isostatic press), and final machined leaving 0.45mm grind stock on the ID. The tubes were shipped to Caterpillar for bench tests described in Tasks 3.3.2, 3.3.3, and 3.3.4. Caterpillar performed the final grinding operation on the valve guides' I.D. per the drawing specifications. Vitreous bonded diamond grinding wheels were used for the I.D. final grinding, according to the procedure developed under Phase I.

For engine testing in the 3500 series natural gas-fueled engine, fifty-six tubes, twenty with no carbon fiber and thirty-six with 10% by volume of carbon fiber, were prepared as described in the previous paragraph by the baseline process. Examples of the guides, containing 10% carbon fiber, are shown in Figure 3.3.1.2-1. Again, Caterpillar final machined the I.D. to the final dimensions and surface finish prior to installing the guides in the test engine under Task 3.4.3. Also fabricated and shipped to Caterpillar were twelve valve guides with 10% carbon fiber for a model 3126 diesel-fueled engine test. The 3126 engine test was not conducted, as explained in Task 3.4.1 Engine Selection.

Figure 3.3.1.2-1. Examples are shown of the CFCC valve guides fabricated under Task 3.3.1.2 . The guides were final ground on the I.D. and engine tested under Task 3.4.4.1 at Caterpillar.

3.3.2 Wear Coefficient

Caterpillar was to generate up to six average wear coefficients for AISI 4140 (Rc50) valve stems running against quarter sections cut from two of the prototype carbon fiber reinforced silicon nitride valve guides for Task 3.4.2 Final Design. In addition, Caterpillar was to generate average wear coefficients for the currently used 3516 S.I. engine series valve stem and guide material pairs, using the procedures outlined in Task 3.2.2. These average wear coefficients were to be used to benchmark the current exhaust valve guide operating performance in this application. Upon completion of this testing, this wear data was to be analyzed to determine if the prototype CFCC exhaust valve guides meet all the tribological performance criteria.

The original specimens received from AlliedSignal had a bore diameter of 9.0mm as called out on the print. However, the I.D. grinding of the bores of the CFCC valve guide blanks took more time than originally expected because the creep-feed grinding techniques developed in Phase I could not be used due the large stock removal. The 92mm bore length required special quills to be designed and fabricated. These quills required that the forces during grinding be kept low to prevent the quills from deflecting during the grinding. Deflection of the quills during grinding would cause excessive taper in the I.D. dimension and very high grinding wheel wear. For this reason, new I.D. grinding techniques had to be developed to reduce the grinding forces. These new

techniques used smaller depth of cuts, which significantly increased the time required to machine the bores.

Therefore, the amount of stock remaining on the bores was reduced. No more than 0.05mm of stock should be removed from the I.D. during this operation (Note -- some machining of the I.D. is required to obtain the required tribological properties).

The modified valve guide specimens were machined and several changes made to the Cameron-Plint test fixture. Testing was then completed for valve guide specimens consisting of monolithic silicon nitride and silicon nitride plus 10 volume percent carbon fiber (no tests were run on cast iron guides). Tests were run against valve stem sections (4140, chrome coated stems). Figure 3.3.2-1 shows the test specimen and fixture with the conditions listed in Table 3.3.2-1.

Figure 3.3.2-1. Valve Stem (left) and Guide Section (in fixture, right) used in the Cameron-Plint Test

Table 3.3.2-1. Cameron-Plint Test Conditions

Temperature	120° C
Lubrication:	1. Dry (with small amount of oil added prior to the test simulating oil present during engine assembly) 2. Lubricated
Load	20N (approx. 20X operating conditions)
Cycles	1.44E06 (8 hours) @ 50Hz

The wear on each valve stem was determined by measuring the wear scar using 3-D surface tracing techniques. Wear on the valve guides was not reported due to the chipping that occurred on the guide edges where the stem impacted during testing. The results of this testing are contained in Table 3.3.2-2.

Table 3.3.2-2. Cameron-Plint Friction and Wear Test Results

Valve Guide Composition	Lubricant	Average Friction Coefficient	Valve Guide Wear Factor	Valve Stem Wear Factor
CFCC/10% carbon fiber	Lubricated	0.12	2.29E-09	None measurable
CFCC/10% carbon fiber	Non-lubricated	0.1	None measurable	None measurable
Monolithic Silicon nitride	Lubricated	0.12	4.5E-08	None measurable
Monolithic Silicon nitride	Non-lubricated	0.16	7.78E-07	None measurable

The results showed that no measurable valve stem wear occurred irrespective of the valve guide composition (monolithic silicon nitride or CFCC) or lubrication condition (lubricated or non-lubricated). However, the average friction coefficient and valve guide wear for the non-lubricated CFCC was slightly lower than that of the non-lubricated monolithic silicon nitride. The lower friction coefficient and valve guide wear factor of the CFCC should result in greater durability in non-lubricated conditions.

3.3.3 Mechanical Property

GS-44 silicon nitride as-densified tube specimens containing 0, 10, and 12.5 volume percent P-75 graphite fiber were supplied by AlliedSignal for mechanical testing. These specimens were machined into Military Std. 1942a, "B" sized modulus of rupture specimens for 4-point flexure testing. The results of this testing are contained in Table 3.3.3-1.

**Table 3.3.3-1: Mechanical Properties of GS-44 Silicon Nitride Densified Tubes
containing various amounts of Amoco P-75 Carbon Fibers**

Fiber Loading (Volume %)	Number of Specimens	Avg. Flexural Strength (Mpa)	Flexural Strength Range (Mpa)	Elastic Modulus (Gpa)	Weibull Modulus
0	20	784	708-826	272	31
10	34	282	231-312	230	17
12.5	30	253	206-307	206	12

The results listed in Table 3.3.3-1 showed that the addition of the carbon fibers to the GS-44 silicon nitride reduced the average flexural strength from 784 to 282 or 253 MPa for the 10 and 12.5 percent volume loadings, respectively (Approximately a 64 and 68 percent decrease in strength). In addition, the Wiebull modulus for these specimens decreased from 31 to below 20.

Scanning electron microscope analyses of the fracture surfaces of the specimens after the flexural testing showed several 50-100 μ m carbon fiber agglomerates. Examples of the fiber agglomerates are shown in Figures 3.3.3-1 and 3.3.3-2.

Figure 3.3.3-1. Silicon Nitride with 10 volume percent Carbon Fiber – 231 MPa breaking stress; SEM MOR bar fracture surfaces at 100x and 500x.

Figure 3.3.3-2. Silicon Nitride with 12.5 volume percent Carbon Fiber – 234 MPa breaking stress; SEM MOR bar fracture surfaces at 100x and 500x.

3.3.4 Durability Test

3.3.4.1 Baseline Test

The objective of this task was to determine the durability of the CFCC material system developed under Phase I. The durability testing was performed on a diesel engine valve stem/valve guide test jointly developed by Caterpillar and Battelle. The bench test is illustrated in Figure 3.3.4.1-1. In general, the test consisted of a single 3500 Series Engine cylinder head with valve train. The cylinder head was mounted to a machine base and an electric motor and flywheel used to rotate the camshaft to operate the valves. In addition, upper and lower enclosures were used to confine the diesel exhaust environment and drip-fed lubricant. Caterpillar provided all the standard production parts to build the test fixture including a head group assembly, four push rods and lifters, camshaft, valve bridge, inlet and exhaust valve rockers, rocker shaft, valve cover, and spacer between the valve cover and cylinder head. A heater was installed in place of a fuel injector and the cylinder head filled with a heat conducting fluid. A temperature of 135°C (270°F) was maintained during the tests.

The test plan's first objective was to determine whether measurable wear could be detected running production hardware (cast iron guides) in the bench test. Once this baseline was established, different configurations of GS-44 silicon nitride and CFCC guides were run in various lubrication conditions.

Figure 3.3.4.1-1. Battelle Durability Test – Motorized 3500 Series Cylinder Head.

To establish the baseline, a 100 hour test was run using cast iron guides with lubrication. The valve guide bores and stem diameters were measured in three locations (relative to the guide) before and after testing: at the center, and at 7mm from the top and bottom. All inner diameter measurements were the average of two readings made 180 degrees apart with a 3-shoe bore micrometer. Stem diameters were made in similar manner with a micrometer. Measurement accuracy was reported to be $\pm 2\mu\text{m}$. The results of the 100 hour baseline, listed in Table 3.3.4.1-1, show that measurable wear was detectable within 100 hours of testing.

Measurable wear was detected in the short hour baseline test. However, the greatest valve guide wear typically occurs at the bottom of the guide. In this case, the

greatest wear was consistently near the top. It should also be noted in hindsight that standard valve guide wear is measured 12.5mm from top and bottom. This will be discussed later with the engine test results. Lastly, the bench test was run at a uniform temperature, with no temperature differential between the guide and stem as in the real engine application that would change the clearance between the guide and stem from differential expansion. Again, this is discussed further with the engine test results.

Table 3.3.4.1-1. Valve Guide and Stem Wear from 100 hour Baseline Durability Test.

Valve Number	Location	Valve Guide ID Wear (μm)	Valve Stem OD Wear (μm)
1	7mm from top	21.2	8
	Center	6.9	0
	7mm from bottom	14.8	18
2	7mm from top	25.7	0
	Center	9.3	0
	7mm from bottom	14.3	3
3	7mm from top	32.3	5
	Center	12.8	3
	7mm from bottom	3.9	5
4	7mm from top	19.2	8
	Center	9.8	8
	7mm from bottom	9.7	8
Averages	7mm from top	24.6	5.3
	Center	9.7	2.8
	7mm from bottom	10.7	8.5

3.3.4.2 Lubricated Durability Tests

Following the baseline test, guides for Test 1 were selected consisting of the production cast iron, monolithic silicon nitride (GS-44), CFCC with 10 volume percent carbon fibers (CFCC/10) and CFCC with 12.5 volume percent carbon fibers (CFCC/12.5). Caterpillar provided a procedure for press fitting the guides into the cylinder head. Wear measurements were taken at 100 and 200 hours of testing. The cast iron guide demonstrated similar wear to the prior baseline test and the cast iron wear was higher than that of any other guide. The stem wear for the GS-44 was the highest, however, the CFCC exhibited no stem wear.

No increase in guide or stem wear was seen in the second 100 hours of testing. This indicates that a 100 hour break-on period may exist for all the guide materials. With the minimal wear in the GS-44 and CFCC guides, they may not have “broken-in” during

this test. After the initial break-in, no additional wear occurred within the test. The 100 and 200 hour measurement results of Test 1 are shown in Table 3.3.4.2-1.

Table 3.3.4.2-1. Valve Guide and Stem Wear after 100 and 200 hours of Durability Test 1 (Lubricated).

Valve Guide Material	Location	Guide ID: 100 hour Wear (μm)	Guide ID: 200 hour Wear (μm)	Stem OD: 100 hour Wear (μm)	Stem OD: 100 hour Wear (μm)
Cast Iron	7mm from top	38.0	37.0	5.1	5.1
	Center	18.5	17.0	----	----
	7mm from bottom	17.5	17.5	6.4	6.4
GS-44	7mm from top	4.5	2.5	7.6	6.3
	Center	0.0	0.0	----	----
	7mm from bottom	7.5	2.5	2.5	1.3
GS-44	7mm from top	7.0	6.0	8.9	7.6
	Center	6.5	2.0	----	----
	7mm from bottom	0.0	2.0	1.3	1.3
CFCC/12.5 percent Carbon Fibers	7mm from top	4.0	5.0	0.0	2.5
	Center	8.0	4.0	----	----
	7mm from bottom	0.0	1.5	0.0	0.0

A second durability test (Test 2) was conducted using a cast iron, two CFCC/10 and a CFCC/12.5 guide, under lubricated conditions. The results were similar to Test 1 in that the cast iron guides wore the most. The CFCC/12.5 and GS-44 guides exhibited slightly more wear in this second lubricated test. However, there was essentially no difference in stem wear between all the guide materials, regardless of the carbon fibers. Also, there was no additional wear in the second 100 hours of test. Table 3.3.4.2-2 shows the 100 and 200 hour wear data for Test 2.

Table 3.3.4.2-2. Valve Guide and Stem Wear after 100 and 200 hours of Durability Test 2 (Lubricated).

Valve Guide Material	Location	Guide ID: 100 hour Wear (μm)	Guide ID: 200 hour Wear (μm)	Stem OD: 100 hour Wear (μm)	Stem OD: 100 hour Wear (μm)
Cast Iron	7mm from top	26.0	26.0	3.2	3.8
	Center	10.5	10.5	----	----
	7mm from bottom	16.5	17.5	2.5	2.5
GS-44	7mm from top	3.0	3.5	1.9	2.5
	Center	3.0	5.0	----	----
	7mm from bottom	12.5	8.0	1.3	1.3
GS-44	7mm from top	6.0	5.0	0.0	0.0
	Center	6.0	3.0	----	----
	7mm from bottom	5.0	5.0	1.9	1.9
CFCC/12.5 percent Carbon Fibers	7mm from top	6.3	2.5	1.9	1.9
	Center	3.0	0.0	----	----
	7mm from bottom	11.5	9.5	1.3	1.3

3.3.4.3 Restricted Lubrication Durability Tests

The Battelle bench test was modified to restrict lubrication of the valve guide and stem to evaluate the full benefit of the carbon fibers in reducing wear. This was accomplished by the use of a rubber "umbrella" valve stem seal placed on the top of the valve guide. Approximately 3000 ml of oil leaked past the guides during the lubricated 200 hour tests without the valve stem seals. Using the rubber "umbrella" stem seals, only 400 ml of oil leaked past the valve guide during Test 3 and approximately 1050ml leaked during Test 4. (Because of the variation in combined leakage between Test 3 and 4, it is likely that there is substantial variation in the leakage between the individual valve stem seals.)

Table 3.3.4.2-3 contains the valve guide and stem wear measurements from the first test with restricted lubrication (Test 3). These results show that the cast iron guide experienced the highest wear. However, after 100 hours, none of the guides showed additional wear. This is unexpected since the lubrication was restricted and this should have impacted the cast iron wear at the very least. Valve stem wear was not significantly different between any of the guide materials or lubrication conditions.

Table 3.3.4.2-3. Valve Guide and Stem Wear after 100 and 200 hours in Durability Test 3 (Restricted Lubrication).

Valve Guide Material	Location	Guide ID: 100 hour Wear (μm)	Guide ID: 200 hour Wear (μm)	Stem OD: 100 hour Wear (μm)	Stem OD: 100 hour Wear (μm)
Cast Iron	7mm from top	23.0	27.5	5.1	7.0
	Center	10.0	9.5	----	----
	7mm from bottom	17.5	14.5	1.9	3.8
GS-44	7mm from top	0.0	0.0	3.8	3.8
	Center	3.0	6.0	-----	----
	7mm from bottom	6.5	7.0	2.5	2.5
CFCC/10 percent Carbon Fibers	7mm from top	4.5	0.0	6.4	7.0
	Center	1.5	3.0	----	----
	7mm from bottom	6.0	2.0	2.5	2.5
CFCC/12.5 percent Carbon Fibers	7mm from top	6.0	5.0	5.1	7.0
	Center	2.5	3.0	----	----
	7mm from bottom	2.5	5.0	3.8	5.1

A second test (Test 4) was conducted with restricted lubrication. The valve guide and stem wear measurements from Test 4 are shown in Table 3.3.4.2-4. As mentioned previously, 1050ml of oil was collected from this 200 hour test versus the 400ml collected from Test 3. The additional oil may have impacted the wear versus Test 3 (restricted). However, Test 4 exhibited the lowest cast iron wear of any of the tests – even lower than Tests 1 and 2 where more oil was used.

Table 3.3.4.2-4. Valve Guide and Stem Wear after 200 hours in Durability Test 4 (Restricted Lubrication).

Valve Guide Material	Location	Guide ID: 200 hour Wear (μm)	Stem OD: 200 hour Wear (μm)
Cast Iron	7mm from top	12.0	1.7
	Center	6.5	----
	7mm from bottom	9.5	0.6
GS-44	7mm from top	0.0	1.1
	Center	2.0	----
	7mm from bottom	0.0	0.6
CFCC/10 percent Carbon Fibers	7mm from top	0.0	1.1
	Center	0.5	----
	7mm from bottom	3.5	0.2
CFCC/12.5 percent Carbon Fibers	7mm from top	0.0	2.8
	Center	2.0	----
	7mm from bottom	0.0	0.6

3.3.4.4 Durability Test Summary

The significant points from the Battelle testing are:

1. Measurable cast iron valve guide wear occurred within 100 hours. This wear is most likely break-in wear.
2. No additional guide wear occurred after 100 hours of testing (with a 200 hour test length) for any of the guide materials or lubrication conditions. Test durations greater than 200 hours would be needed to truly evaluate the guide materials in this bench test.
3. Stem wear is insignificant regardless of test duration (up to 200 hours), guide material or lubrication condition.
4. Variability exists in lubrication as evidenced by the different amounts of oil collected for each test.
5. Wear measurements were taken 7mm from the valve guide ends (and matching location on the valve stem). Standard guide measurements for engine testing are taken 12.5mm from each end. In later sections, it will be shown that the guide ID becomes bell shaped near each end during “break-in”. By measuring only 7mm from the end, only the break-in may have been measured.

Task 3.3.5 Selection of CFCC Valve Guides

CFCC guides with 10 volume percent carbon fibers were selected for additional testing. The volume percent was selected to minimize the amount of carbon fiber and cost without impacting the guides functionality since no difference in wear was observed in the Battelle test between 10 and 12.5 percent carbon fibers.

3.4 Component Evaluation (Task 3.4)

3.4.1 Engine Selection

The engine selected for testing was a 3508 Natural Gas Engine. The engine specifications are listed in Table 3.4.1-1 and a picture of the engine is shown in Figure 3.4.1-1. The original engine targeted in the program was a low-heat-rejection (LHR) engine that would operate with valve guide temperatures above approximately 260°C. At such temperatures, oil “cokes” and causes valve sticking. Therefore, a guide would be required that could operate without oil lubrication, hence, the rationale for using the carbon fiber for lubrication. Since the LHR engine was not available, the natural gas engine was selected. The engine has a production configuration and would also benefit from self-lubricating valve guides, because it lacks the lubrication produced during diesel fuel combustion. However, since the natural gas engine operates at guide temperatures much lower than the LHR engine, the benefit of high-temperature lubrication was not evaluated in this engine test.

A 3600 series engine was also considered for testing. However, the size of this guide was beyond current processing capabilities at AlliedSignal. A 3126 Series engine was selected as the second test bed, following successful completion of the 3500 Natural Gas engine test. Guides were produced and machined for this engine. However, the guide design changed with an engine modification during the time the guides were produced and the 3500 engine test was conducted. Therefore, these guides could not be engine tested.

Table 3.4.1-1. 3508 Series Natural Gas Engine Specifications

Speed	1500 rpm
Brake Mean Effective Pressure	1240 kPa
Timing	24 degrees
Emissions Level	180ppm NO _x (TA LUFT or 1.1 g/bhp-hr.)
Guide Temperatures	100 to 150°C
Valve Head Temperatures (approximate)	Intake 300°C Exhaust 550°C

Figure 3.4.1-1. 3508 Natural Gas Engine Installed in Test Cell

3.4.2 Final Design

The final valve guide design used for the 3508 engine testing is shown in Figure 3.4.2-1. The design is based on the existing cast iron guide, and is press fitted into the cylinder head. The guide inner diameter and stem outer diameter provide a cold diametrical clearance from 0.082 to 0.040mm. The outer guide diameter was press fit into the cylinder head bore with a press fit ranging up to 0.037mm. Engine test

experience has shown that cast iron guides “close-in” an average of 0.023mm as a result of the press fit.

Figure 3.4.2-1. Final valve guide geometry

3.4.3 Engine Assembly

The inner diameter surface finish and morphology of two CFCC guides and one monolithic silicon nitride guide, all machined at Caterpillar, were examined. The examination consisted of first evaluating the surface finish of the machined bores using 3-D surface analysis techniques, followed by optical and scanning electron microscopy techniques to evaluate the surface morphology and the effect of the machining on the carbon fibers. The purpose of this small study was to quantify the surface condition of the CFCC and monolithic valve bores prior to engine testing

The results of the 3-D surface analysis are listed in Table 3.4.3-1. Also included in Table 3.4.3-1, for comparison, was a 3-D surface analysis of a CFCC bore prior to

machining at Caterpillar. The bores, prior to machining at Caterpillar, had surface roughness (Ra) ranging between 0.664 to 0.668 μm . One reason for the high Ra values was that there were machining lines present. An optical examination of the bores showed that no carbon fibers were present on the machined surface. Machining the bores at Caterpillar reduced the average surface roughness (Ra) to 0.233 or 0.388 μm . An optical examination of these CFCC bores showed that the carbon fibers were not damaged during machining. Further, the surface morphology and amount of carbon fiber damage due to machining did not vary along the length of the machined CFCC bore. The roughness of the monolithic silicon nitride bores, after machining at Caterpillar, ranged between 0.040 to 0.489 μm .

Table 3.4.3-1. Surface Inspection of Machined Valve Guides

Specimen	Location	Ra Value (μm)
CFCC Blank -- as received	Top	0.664
	Bottom	0.683
CFCC Valve Guide 1	Top	0.302
	Bottom	0.388
CFCC Valve Guide 2	Top	0.233
	Bottom	0.302
Monolithic Silicon Nitride	Top	0.489
	Bottom	0.46

3.4.4 Engine Tests

The original test plan was to run the engine for 250 hours and then measure the valve stem and guide wear. Also, valve seat recession was to be measured (excess valve guide to valve stem clearances could cause valve seat wear). If all these measurements were within Caterpillar's engine tolerance specifications, the engine was to be run for an additional 250 hours.

3.4.4.1 Engine Test 1 - 224 Hours

The first engine test began on July 21, 1997 with the valve guide configuration shown in Figure 3.4.4.1-1. No problems were encountered during installation of the guides.

SN = Monolithic Silicon Nitride guides

CFCC = CFCC Guides (10 volume Percent Carbon fibers)

CI = Cast iron guides (Baseline)

1,2,3,4 valve positions in cylinder

Figure 3.4.4.1-1. Valve Guide Configuration for Engine Test 1 – 224 Hours

The test engine had accumulated 224 hours before a facility problem caused the engine to shut down. During the shut down, cylinder head covers were removed and the CFCC valve guides were examined. This examination revealed that 10 of the 16 CFCC and 1 of the 8 monolithic silicon nitride guides moved during the test. Seven of the CFCC guides that moved were pushed out between 10 to 11 mm. The remaining 3 CFCC guides were pushed into the cylinder head. The one monolithic silicon nitride valve guide was pushed out.

Inspection of the guide OD and cylinder bore ID revealed that the ceramic guides were machined to print and the cast iron guides were typically oversized. The cylinder head bores IDs were typically oversized up to 0.2mm, resulting in the valve guide movement. Guides that remained stationary had cold press fits ranging from 0.004 to 0.006mm.

The measurement results for guide and stem wear from the 224 hour test are listed in Tables 3.4.4.1-1 and 3.4.4.1-2. Only guides that did not move were included in the calculations. The results show that the cast iron guides and mating stems did not wear during the test. Conversely, the CFCC/12.5 guides and mating stems wore the greatest, in particular on the bottom exhaust position. The GS-44 guides and mating stems wore more than the cast iron, but about half that of the CFCC/12.5. The GS-44 wear was also greater on the bottom exhaust guides and stems.

Table 3.4.4.1-1. Guide Wear (microns) from Test 1 – 224 Hours

INTAKE						
Guide Material	Avg. Guide Wear-Bottom	Min. Guide Wear-Bottom	Max. Guide Wear Bottom	Avg. Guide Wear-Top	Min. Guide Wear-Top	Max. Guide Wear-Top
CFCC/10.5	4	0	15	1	0	2
GS-44	4	0	12	3	0	7
Cast Iron	1	0	3	2	0	6
EXHAUST						
Guide Material	Avg. Guide Wear-Bottom	Min. Guide Wear-Bottom	Max. Guide Wear Bottom	Avg. Guide Wear-Top	Min. Guide Wear-Top	Max. Guide Wear-Top
CFCC/10.5	22	0	42	1	0	3
GS-44	14	0	32	3	0	6
Cast Iron	0	0	2	3	3	5

Table 3.4.4.1-2. Stem Wear (microns) from Test 1 – 224 Hours

INTAKE						
Guide Material	Avg. Stem Wear-Bottom	Min. Stem Wear-Bottom	Max. Stem Wear-Bottom	Avg. Stem Wear-Top	Min. Stem Wear-Top	Max. Stem Wear-Top
CFCC/10.5	32	10	73	8	0	31
GS-44	5	1	8	0	0	0
Cast Iron	1	0	2	0	0	1
EXHAUST						
Guide Material	Avg. Stem Wear-Bottom	Min. Stem Wear-Bottom	Max. Stem Wear-Bottom	Avg. Stem Wear-Top	Min. Stem Wear-Top	Max. Stem Wear-Top
CFCC/10.5	49	2	108	18	1	32
GS-44	23	1	59	3	0	7
Cast Iron	1	0	4	0	0	2

Task 3.4.4.2 Engine Test 2-528 Hours

A second engine test was planned for the following reasons:

1. Several CFCC guides had moved during the first test.
2. There may not have been enough test time to accumulate significant wear in the cast iron.
3. The break-in period could be longer for CFCC guides.
4. The wear results were not as expected.

Fifteen unused CFCC and silicon nitride guides, and eight replacement cylinder heads were inspected for the additional 500 hours of testing. All of the cylinder heads had valve guide bores that were the correct dimension. Nine of the guides were not useable because of undersized outer diameters and/or oversized inner diameters. Therefore, several conforming guides were selected from the first 224 hour test to be used for continued engine testing. Three CFCC guides failed during installation and were replaced with cast iron guides. The final valve guide test configuration is shown in Figure 3.4.4.2-1.

SN = Monolithic Silicon Nitride guides

CFCC = CFCC Guides (10 volume Percent Carbon fibers)

CI = Cast iron guides (Baseline)

1,2,3,4 valve positions in cylinder

Figure 3.4.4.2-1. Valve Guide Configuration for Engine Test 2 – 528 Hours.

The 500 hour test started running September 22, 1997 with 13 CFCC, 8 silicon nitride, and 11 cast iron guides. Initially, this test was planned to run without lubrication of the valve guides. However, the stem seals used on all guides metered sufficient oil down the valve stem to consider the test fully lubricated. The engine was inspected after approximately 4 hours of operation and no guides had moved.

During a planned inspection at approximately 280 hours, one of the CFCC guides had moved up in cylinder number 3. The entire cylinder head was removed and replaced with one containing all cast iron guides. This reduced the total number of guides to 7 silicon nitride, 12 CFCC, and 13 cast iron. Note that the valve guide bores and guide outer diameters were inspected prior to the engine test. Guide bores were measured in four locations - two at the bore top and bottom. All bores were within print tolerance. Guides were inspected similarly (4 locations on both the inner diameter and outer diameter) and those within print tolerance were used for the engine test. Therefore, the thermal expansion mismatch between the cast iron cylinder head and CFCC/12.5 guide may require a greater press fit.

A second cylinder head (number 1) was replaced near the completion of the 500 hours to accommodate other in-house testing. The engine was then run to 528 hours with 6 of the 8 original cylinder heads. Cylinder heads that completed at least 500 hours were inspected for guide and stem wear with the results tabulated in Tables 3.4.4.2-1 and 3.4.4.2-2. The results of Test 2 were similar to Test 1 in that the CFCC wore the greatest, followed by the GS-44. The wear was primarily at the bottom exhaust positions. The cast iron guides and mating stems exhibited no wear regardless of location.

Table 3.4.4.2-1. Guide Wear (microns) from Test 2 – 528 Hours

INTAKE						
Guide Material	Avg. Guide Wear-Bottom	Min. Guide Wear-Bottom	Max. Guide Wear Bottom	Avg. Guide Wear-Top	Min. Guide Wear-Top	Max. Guide Wear-Top
CFCC/10.5	5	0	29	0	0	0
GS-44	0	0	0	0	0	1
Cast Iron	0	0	0	0	0	1
EXHAUST						
Guide Material	Avg. Guide Wear-Bottom	Min. Guide Wear-Bottom	Max. Guide Wear Bottom	Avg. Guide Wear-Top	Min. Guide Wear-Top	Max. Guide Wear-Top
CFCC/10.5	25	0	71	0	0	0
GS-44	8	0	20	0	0	0
Cast Iron	3	0	17	0	0	1

Table 3.4.4.2-2. Stem Wear (microns) from Test 2 - 528 hours

INTAKE						
Guide Material	Avg. Stem Wear-Bottom	Min. Stem Wear-Bottom	Max. Stem Wear-Bottom	Avg. Stem Wear-Top	Min. Stem Wear-Top	Max. Stem Wear-Top
CFCC/10.5	27	7	37	9	5	18
GS-44	3	1	6	4	1	8
Cast Iron	2	0	8	2	0	8
EXHAUST						
Guide Material	Avg. Stem Wear-Bottom	Min. Stem Wear-Bottom	Max. Stem Wear-Bottom	Avg. Stem Wear-Top	Min. Stem Wear-Top	Max. Stem Wear-Top
CFCC/10.5	160	18	401	39	0	68
GS-44	37	0	92	9	1	21
Cast Iron	1	0	3	1	0	3

3.4.5 Data Analysis/Recommendation

The results from both the 224 and 528 hour tests were consistent in that the CFCC guide and mating stem wore the most. However, no significant wear occurred with the cast iron guide and mating stem. Figures 3.4.5-1 and 3.4.5-2 graphically compare the average wear found in the 224 and 528 hour tests.

Figure 3.4.5-1. Intake and Exhaust Guide Wear for Test 1 and 2.

Figure 3.4.5-2. Intake and Exhaust Stem Wear for Test 1 and 2.

Potential compounding reasons for the high CFCC and silicon nitride wear that were investigated were:

1. Thermal expansion mismatch and stem to guide clearance: The silicon nitride based guides have a lower coefficient of thermal expansion (CTE) which may result in a smaller clearance between the valve stem and guide at engine operating temperatures.
2. Incorrect guide geometry and material break-in differences: The cast iron may "break-in" at the end of the guides, relieving contact stress with the valve stem. Conversely, the ceramic guides may not "break-in" at the guide ends and may have caused line contact between the stem and guide resulting in the adverse wear.
3. Material variations within the silicon nitride guides: Material processing may have caused variability in the material microstructure or carbon fiber distribution which resulted in adverse wear.

3.4.5.1 CTE Mismatch Affecting Guide to Stem Clearance

Thermal expansion mismatch between the silicon nitride or CFCC guide and steel valve stem may have reduced the clearance and resulted in the adverse wear. This theory is supported by the following:

1. Wear was always greater on the bottom of the exhaust valve and guides. The temperature would be the greatest at this location, exaggerating the thermal expansion mismatch.
2. The CTE of the cast iron guide is nearly 3X that of the silicon nitride guide which may result in a smaller clearance with a steel valve stem with the silicon nitride based guides.
3. Prior in-house valve guide testing showed a relationship between guide clearance and wear. If there was insufficient clearance between the guide and stem, the guide would wear until a certain minimum clearance was obtained.
4. The Battelle test was run at a uniform temperature of 135°C. All the components would be at the same temperature. In the engine, the valve guides are cooled by their contact to the water cooled cylinder head. The valve (and stem) are heated by the combustion process creating a large temperature differential between the guide and stem.

Table 3.4.5-1 lists the clearance range versus assumed stem to guide temperature differential and shows that a 10 micron diametrical difference in clearance exists between the cast iron and silicon nitride guides. Also, the valve stem will contact the silicon nitride guides at approximately 100°C lower temperature than the cast iron guides.

Table 3.4.5-1. Clearance change versus temperature and material

	Cast Iron CTE $\approx 10.5E-06/C$	CFCC and Si3N4 CTE $\approx 3.2E-06$
Stem to Guide Temperature Differential (°F)	Diametrical Clearance Range (microns)	Diametrical Clearance Range (microns)
0 (Room Temperature)	15 to 77	15 to 77
60	10 to 72	0 to 62
80	8 to 70	-2 to 60
160	0 to 62	-10 to 52

The thermal expansion difference appears to be a factor in the CFCC and silicon nitride guide wear. Therefore, the clearance between the guide and stem should correlate to the wear. For example, a silicon nitride guide with a large guide to stem clearance should have less wear than one with a low clearance. All the guides and stems were measured before and after the test. The data was examined for both the 224 and 528

hour test to see if there was a correlation between operating clearance (calculated from starting clearance and assumed temperature differential) and wear. The data from Test 1 (the 224 hour test) is compared in Figures 3.4.5.1-1 and 3.4.5.1-2, and the data from Test 2 (the 528 hour test) is compared in Figures 3.4.5.1-3 and 3.4.5.1-4.

The 224 hour test seemed to show a correlation between estimated operating clearance and wear in particular with the CFCC guide which had the smallest clearance and greatest wear. However, the silicon nitride exhaust guide and mating stem did not follow the trend. The 528 hour test only showed correlation between clearance and wear at intake locations. Therefore, thermal expansion mismatch may play a role in the CFCC and silicon nitride wear. However, it is only one of the factors and may only play a minor role.

Figure 3.4.5.1-1. Test 1 – Intake and Exhaust Guide Wear Versus Clearance

Figure 3.4.5.1-2. Test 1 – Intake and Exhaust Stem Wear Versus Clearance

Figure 3.4.5.1-3. Test 2 – Intake and Exhaust Guide Wear Versus Clearance

Figure 3.4.5.1-4. Test 2 – Intake and Exhaust Stem Wear Versus Clearance

3.4.5.2 Geometry and Material Break-in Differences

The ceramic guides have two to three times the elastic modulus of the cast iron guides. The higher stiffness may prevent or change break-in behavior relative to the cast iron. The valve guide inner diameter is machined parallel to the outer diameter, creating the possibility of line contact at the guide top and bottom.

During break-in, the cast iron guides may wear near the bottom, forming a bell-shaped inner diameter so that the line contact or knife edge is blunted. The high stiffness ceramic may maintain the knife edge longer, causing valve stem wear.

The bell-shaped wear that may occur in the cast iron valve guides would not be detected by the standard guide wear measurements since the measurements are taken 12.5mm from either guide end. Therefore, the inner diameter of the valve guides was traced from end to end using a coordinate measuring machine (CMM). Traces were taken 90 degrees apart to give a relative picture of the worn inner diameter profile. The guides were not in the cylinder head during the inspection.

Initial examination of the 224 hour guides using the CMM method showed a difference in guide geometry between materials as shown in Figure 3.4.5.2-1. This illustration of the cast iron guide shows the extent of “banana” shaping of the guide ID. However, there were examples of straighter cast iron IDs. (This shows the variability that exists between guides, even of the same material, that may be dependent on location, engine assembly, and tolerance stack-up).

Figure 3.4.5.2-1. Inner Diameter (ID) trace of 224 hour CFCC (top) and cast iron guide (bottom).

Valve guides from the 528 hour test were also inspected using the CMM machine to determine if geometry differences existed between guide materials. The inspection was done with the guides installed in the cylinder head to determine if there were orientation effects on the valve guide wear, i.e. greater wear in the rocker arm axis. These inspections showed there were examples of straight IDs as well as curved/bell-shaped IDs for all guide materials, similar to the 224 hour traces.

The ID traces show geometry but were not used to measure an average inner diameter. To obtain the actual diameter difference between materials and measurement location (bottom versus 12.5mm from bottom), diameter measurements were taken with a dial bore indicator (used for prior wear measurements). An illustration of the two measurement locations is shown in Figure 3.4.5.2-2. The results of the measurements are listed in Table 3.4.5.2-1.

Figure 3.4.5.2-2. Valve Guide Measurement Locations For Determining Wear at Guide Bottom

Table 3.4.5.2-1. Inner Diameter Measurements of 528 Hour Hardware

Material	Location	Average Diameter Difference (μm)	Number of Guides Measured
Cast Iron	New	10	1
	Exhaust	50	6
	Intake	16	3
CFCC	New	-25	1
	Exhaust	2	2
	Intake	0	3
Si3N4	New	5	1
	Exhaust	23	1
	Intake	0	1

Based on the limited number of guides available for measurement, it appears that cast iron guides wear more at the bottom than the silicon nitride or CFCC guides (there is

less differential between the measurement locations for both CFCC and silicon nitride). However, from prior tables, the cast iron shows no wear at 12.5mm in from the guide end. Machining procedures were developed that allowed the CFCC and silicon nitride valve guides to be machined with a tapered inner diameter. Essentially, the deflection of the quill provided an acceptable taper, increasing the diameter by approximately 80 microns on each end. This procedure could be easily adapted to machining guides and is actually an artifact that has to be carefully removed to make the guide diameter straight.

It should be noted that the CFCC exhaust guides showed an average wear of 25 microns at 12.5mm from the bottom. If the CFCC guide IDs became "bell-shaped" early in the engine test, they may have worn "straight" during the latter portions of the test. A similar possibility exists with the silicon nitride guides which wore an average of 8 microns (exhaust location) during the 528 hour test.

3.4.5.3 Material Variation

Material processing may have resulted in variability in the material microstructure or carbon fiber distributions resulting in adverse wear. To examine this possibility, GS-44 and CFCC guides from the 528 hour test were selected. Examples of low and high wear guides were sectioned for qualitative examination in a SEM. Essentially no microstructural differences could be seen when examining the center portions of the guide where no wear had occurred. However, there were clear differences between wear locations within each guide. These differences are illustrated in Figures 3.4.5.3-1 and 3.4.5.3-2.

Figure 3.4.5.3-1. Microstructure of CFCC valve guide inner diameter. Top: Unworn and as-machined CFCC surface with several machining lines present. Middle: “Medium” wear view taken from approximately 12.5mm from the guide bottom showing discrete fiber cross sections (dark phase). Bottom: “High” wear view taken from the bottom inner diameter where severe wear had occurred. Some carbon fibers still present, voids left as well.

Figure 3.4.5.3-2. Microstructure of silicon nitride valve guide inner diameter from the 528 hour engine test. Top: Unworn and as-machined silicon nitride surface with several machining lines present. Middle: “Medium” wear view taken from approximately 12.5mm from the guide bottom showing removal of machining marks. Bottom: “High” wear view taken from the bottom inner diameter where severe wear had occurred. The high wear area appears highly polished.

3.4.6 Expanded Bench Testing

The objective of this task was to determine the role and performance of carbon fibers in the CFCC valve guides compared to GS-44 silicon nitride and cast iron in controlled bench tests. Results of the engine testing were not expected and controlled tests were planned to study the material behavior outside the influence of the engine. Two types of bench tests were conducted under different lubrication, loading, time and geometry conditions and the resulting friction coefficients and surfaces analyzed. The surface examination was conducted to determine if a carbon film forms and if it is consumed in the duration of test.

The first bench test was conducted at Oak Ridge National Lab (ORNL) using a “ball on flat” configuration. The second bench test was conducted at Caterpillar using actual guide and stem sections in a modified Cameron-Plint test.

The ORNL ball on flat bench test was conducted on flat sections of all three valve guide materials. Valve guides (taken from engine tested hardware) were machined into flat sections with the same surface finish as the guide inner diameter. A 440C ball was run perpendicular to the machining direction at a velocity equivalent to the average engine valve velocity. Tests were run in both lubricated and unlubricated conditions with different loads and durations to determine the resulting friction coefficients. The surfaces were qualitatively and studied for formation and consumption of the carbon. Wear rates were also determined.

The Caterpillar Cameron-Plint test was modified to accept a valve stem section and to slide it back and forth inside a 14mm length of a valve guide (Figure 3.4.5-1). Different loads, lubrication, and temperature conditions were used in the overall test matrix. Selected surfaces were qualitatively examined.

Figure 3.4.6-1. Cameron-Plint Test Fixture Schematic for Holding Valve Guide and Stem

3.4.6.1 ORNL Ball-on-Flat Testing

The following is the executive summary from ORNL's final report, which is attached in its entirety in Appendix A.

Reciprocating ball-on-flat tests were conducted on prepared sections of cast iron, silicon nitride, and silicon nitride/10 vol. % graphite Chopped Fiber Ceramic Composite (CFCC) valve guides over a range of temperatures, normal loads, speeds, and lubrication conditions. The purpose of this work was to ascertain whether the latter ceramic composite would produce a beneficial lubricating film on the valve guide bore surface and/or the valve stem. Type 440C stainless steel was used to simulate the valve stem material. Machining practices were selected to provide the surface roughness and lay (direction of grinding marks) similar to that of actual valve guide bores. For comparison with the ceramic composite material, both cast iron and silicon nitride matrix materials were also tested. Tests were also performed using graphite powder on the silicon nitride matrix material to simulate what frictional behavior might be observed in the most favorable case.

Friction and wear data, combined with surface chemical analysis confirmed that the current composite, while wear resistant, did not provide any lubrication advantages over silicon nitride itself. No evidence for the sliding-induced formation of a beneficial graphite film was obtained by optical examination, scanning electron microscopy, or surface chemical analysis. While the present CFCC did not provide the needed lubricating effect, the results do not preclude the possibility of developing other ceramic composites with self-lubricating properties.

3.4.6.2 Caterpillar Cameron-Plint Testing

The objective of this test was to evaluate the valve guide material performance separately from the design influences discussed in Section 3.4.5 using actual engine components. The two primary design influences discussed were thermal expansion mismatch and break-in differences. These influences were corrected by enlarging the guide inner diameter from 9.502 to 9.602mm and adding a radius at the guide ends. The larger diameter should eliminate thermal expansion mismatch to close stem to guide clearance. Adding the radius at the guide ends should prevent severe line contact between the stem and guide as the stem slides in and out of the guide.

The test conditions are given in Table 3.4.6.2-1. Pictures of the test fixture and Cameron-Plint machine are shown in Figure 3.4.6.2-1.

Table 3.4.6.2-1. Cameron Plint Test Conditions

Load	20 N
Frequency	20 Hz
Duration	2 hours or 1.5 friction coefficient
Lubrication	Dry or Lubricated
Temperature	150 C (dry) or 175 C (lubricated)

1. Full Apparatus

2. Upper Carriage

3. Lower Carriage

4. Carriage Assembly

Figure 3.4.6.2-1. Cameron-Plint Bench Test

All specimens were inspected for surface finish and dimensional accuracy prior to testing. Lubricated tests were performed first and tests were run to completion (2 hours) without difficulty. Friction coefficients for the lubricated test were similar to those reported by ORNL and are listed in Table 3.4.6.2-2. Figure 3.4.6.2-2 shows the minimal wear scar on the valve stems after the bench tests. No wear scars were evident on the guide IDs, therefore, cross sections were not made.

The tests performed without lubrication resulted in friction coefficients that exceeded 1.5 and caused the test to shut down. Examination of the wear scars (shown in Figure 3.4.6.2-3) showed that alignment was critical in this test and was not achieved. Several attempts were made to correct the problem since no friction coefficients had been reported that went above 1.5. Therefore, the friction coefficients of the dry tests proved inconclusive.

Table 3.4.6.2-2. Friction Coefficient Results for Cameron-Plint Bench Test - Lubricated

Material	Friction Coefficient
Cast Iron	0.154
Silicon Nitride	0.147
CFCC	0.140

Figure 3.4.6.2-2. Wear Scars on Valve Stem and Guide after Testing - Dry.

Figure 3.4.6.2-3. Wear Scars on Valve Stem and Guide after Testing - Lubricated.

4.0 Summary of Phase II Accomplishments

A method to fabricate CFCC valve guides was developed and demonstrated. Initially, two CFCC material formulations (10% and 12.5% by volume carbon fiber) were evaluated in mechanical property tests. The 10% by volume formulation was shown to provide adequate lubrication. A baseline valve guide fabrication process was developed, using cold isostatic pressing to form the guide blanks and hot isostatic pressing to densify the guide blanks. Valve guide blanks were fabricated for durability tests in a diesel engine rig. Caterpillar selected a 3500 series engine, using natural gas fuel, for engine testing. Valve guides (CFCC, silicon nitride, and cast iron) were final machined per Caterpillar specifications and tested in the 3508 engine.

While engine test preparations were made and engine tests were being conducted, refinements were made to the valve guide fabrication process. The refinements increased the fabrication process yields and minimized the amount of grind stock to be machined from fully densified valve guide blanks.

The cumulative engine test time exceeded 750 hours. The engine test results contradicted the earlier bench and rig test results, in that the wear was higher in CFCC valve guide/stem pairs from the engine test. The engine test result prompted additional bench and rig tests. Caterpillar and Oak Ridge National Laboratory conducted the tests and analyzed the results, concluding that no advantage in wear resistance is provided by the CFCC valve guide.

Based on the unfavorable results, it is concluded that the work planned for Phase III, pilot scale process development, is not warranted.

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