EFFECTS OF CERAMIC SURFACES ON AUTOIGNITION IN A CONSTANT VOLUME COMBUSTION APPARATUS

INTERIM REPORT

BFLRF No. 246

By

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A constant volume combustion apparatus was used to determine the effects of ceramic-coated surfaces on the ignition of fuel sprays from a diesel injection nozzle. The goal was to determine if the ceramic surfaces could enhance ignition through catalytic effects on the surfaces. Statistical analysis of the data indicated that an aluminosilicate coating had no effect, while a partially stabilized zirconia increased the delay times. It appears, however, that the aluminosilicate coating could actually enhance the ignition, producing shorter ignition delay times, if the results of the appropriate baselines are considered.
FOREWORD

This work was performed at the Belvoir Fuels and Lubricants Research Facility (BFLRF) located at Southwest Research Institute (SwRI), San Antonio, TX, under Contracts No. DAAK70-82-C-0001, DAAK70-83-C-0007, and DAAK70-87-C-0043, for the period 16 February 1984 through 31 December 1987. Work was funded by the U.S. Army Foreign Science and Technology Center (FSTC), Charlottesville, VA, and the U.S. Army Belvoir Research, Development and Engineering Center (Belvoir RDE Center), Ft. Belvoir, VA. Mr. F.W. Schaekel, Belvoir RDE Center (STRBE-VF), was the Contracting Officer's Representative, and M. Scott Mingledorff and Steve Miller were the technical monitors at FSTC/AIAYST-RA-ST3.
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I. INTRODUCTION

Application of ceramic materials in the combustion chambers of diesel engines offers several opportunities for improved cycle efficiency and reduced engine maintenance. The ceramics, primarily in the form of coatings, have been examined mainly as a means of insulating the engine and thus eliminating the need for engine cooling. The elimination of the cooling system has the potential of simplifying the engine maintenance and of providing an opportunity to recover energy via the use of turbo-compounding. Theoretically, the higher combustion chamber temperatures could also result in improved cycle efficiency due to the higher initial temperatures of the expansion stroke. The thermal effects on efficiency have not, however, been definitively demonstrated in an engine.

An intuitive impression is that the ceramic surfaces in the combustion chamber could act as catalysts, which could affect the ignition and combustion of the injected diesel fuel. This impression has been somewhat enhanced by the discussion in Reference 1 and the patent of Morotsky, et al.(2)* In these studies, it is suggested that the coatings act as catalysts, which enhance the ignition and speed combustion. The global outcomes of these effects are improvement in cold starting and reductions in gum and carbon deposits in the engines.

An underlying consideration of the catalytic theory of the ceramic coatings is the actual importance of the surfaces on ignition and combustion in diesel engines. In addition, if the surfaces are indeed acting as catalysts, it appears to be essential that the fuel have direct contact with the combustion chamber surfaces. Examination of the effects of various surfaces on ignition and combustion in an actual engine is extremely difficult due to the problems associated with lube oil and combustion product contamination, component durability, and the problem of detecting subtle changes in combustion.

Documentation of the effects of ceramic surfaces on ignition in a diesel engine is a complex problem involving not only the uncontrollable parameters mentioned above, but also the composition of the coating and the geometry of the injection system and the combustion chamber. The problem therefore reduces to one of designing an experiment

* Underscored numbers in parentheses refer to the references at the end of this report.
in which the uncontrollable parameters are eliminated as variables and the controllable parameters are easily fixed, or at least understood.

An extensive research and development effort has been underway at Southwest Research Institute for the past 3 years aimed at developing a better understanding of the ignition process in diesel engine type environments. These experiments have been performed in a constant volume combustion apparatus (CVCA). Because of the experience in studying ignition in the CVCA and because of its simple geometry, the CVCA appears to be an excellent apparatus for examining the effects of the ceramic surfaces on ignition.

II. OBJECTIVE

The objective of this study was to determine the effects of various ceramic surfaces on ignition delay time as measured in a constant volume combustion apparatus.

III. EXPERIMENTAL

A. Apparatus

In an effort to simplify the experimental problems and to maximize the benefits of utilizing experience gained in previous projects, a series of experiments were performed in an existing constant volume combustion apparatus. The CVCA is currently being developed as a device for determining the cetane number of diesel fuels.(3,4)

The CVCA consists of a high-pressure, high-temperature combustion bomb equipped with a diesel injection system and a sensitive pressure-measuring system. Fig. 1 is a simplified cross-sectional view of the bomb. The apparatus consists of a stainless-steel cylinder machined internally to prevent fuel impingement on the metal surfaces when using a pintle nozzle. The internal volume is 52.3 cubic centimeters with a diameter of 2.5 cm and an approximate length of 10 cm. The spray characteristics of the nozzle have been well documented with respect to penetration, spray angle, and drop size. This type of injection nozzle was chosen because it produces (5) a more dispersed spray in a quiescent environment than a multihole nozzle.
The combustion bomb can be charged with any gaseous oxidizer or inert gas, but the design limits were based on an initial pressure and temperature of 4.1 MPa (600 psia) and 538°C (1000°F), respectively, with a stoichiometric mixture of diesel fuel and air.

The theoretical peak pressure corresponding to the design condition is approximately 34.9 MPa (5000 psia). The bomb is equipped with electrical resistance heaters that are used to heat the vessel walls as well as the contents of the combustion chamber. An automatic controller is used to control the surface temperature of the bomb. Surface thermocouples and a thermocouple probe are used to determine the temperature of the bomb walls and contents, respectively. A water-cooled piezoelectric pressure transducer is installed for measurement of the pressure-time relationship. A charge amplifier is used to condition the transducer signal.

The injection nozzle is equipped with a line pressure transducer installed at the nozzle and a needle-lift sensor. The fuel injection system, specially developed for single-shot injection, has been designed to develop injection-system dynamics and characteristics similar to those observed on systems installed on engines. The injection system consists of a barrel-and-plunger assembly mounted in a pneumatically actuated drive system. As indicated by Hurn, et al. (6,7), the ignition delay times are greatly affected by the rate of fuel injection. Considerable effort was therefore devoted to the development of the
injection system. The primary concern was the repeatability of the injection event not only when using fuels with similar physical properties (viscosity, gravity, and surface tension) but also with fuels of broadly varying physical properties.

The chamber pressure data and the line-pressure and needle-lift data are displayed digitized, and stored on floppy disc using a high-resolution Nicolet digital oscilloscope. An interface installed between the digital oscilloscope and a Hewlett-Packard 1000F computer is used to transfer data from the scope to the computer for computations and permanent storage.

The CVCA was fitted with inserts that could be coated with the various ceramics. These inserts were machined in such a manner that the inserts formed the inside walls of the cylindrical combustion chamber. Three inserts were prepared for testing in the CVCA. One was uncoated for use in establishing the baseline, one was coated with gamma alumina and amorphous silicon oxide, and the third was coated with a yttria containing partially stabilized zirconia (PSZ). TABLE 1 is a listing of the coatings and the treatment procedures.

<table>
<thead>
<tr>
<th>Coating Material</th>
<th>Treatment Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>None</td>
</tr>
<tr>
<td>Aluminosilicate on Molybdenum Bond Coating</td>
<td>Molybdenum (0.003 in.) (0.076 mm)</td>
</tr>
<tr>
<td></td>
<td>Y-Alumina + Amorphous Silica (0.003 in)</td>
</tr>
<tr>
<td>Yttria Containing Partially Stabilized Zirconia</td>
<td>Amdry 995 (0.005 in. thick) (0.127 mm)</td>
</tr>
<tr>
<td></td>
<td>Metco 204 NS (0.012 in. thick) (0.305 mm)</td>
</tr>
</tbody>
</table>

B. Preparation of Coatings

The objective was to apply an aluminosilicate coating similar to the coating generally described in Reference 2. The major claim for this coating is that, when applied to diesel engine combustion chamber, it promotes combustion at high loads and prevents formation of gummy residues at lower loads. The composition of the coating is at least
40 wt% silica with the remainder being predominantly gamma-alumina. The ceramic coating is applied over an intermediate layer of molybdenum. The molybdenum sublayer serves as "bond" coating that damps the differences in thermal expansion of the ceramic and the substrate metal in case of heating and cooling cycles. The molybdenum is also thought to intensify the catalytic activity of the aluminosilicate coatings when applied by powder flame spray method.

Various attempts were made to deposit aluminosilicate coatings on molybdenum-layered stainless steel surfaces. The powder feeds for the plasma-spray gun included premixed alpha-alumina/quartz silica, gamma-alumina/quartz silica, and simultaneous dual feed of the pure components. The variation of spray deposition parameters included nozzle size (powder feed rates), carrier gas mixture (nitrogen and hydrogen), and plasma current and voltage. The coatings obtained by these procedures did not contain significant levels of silica.

The procedure that yielded the required levels of silica (~40 wt%) was spraying pure amorphous silica over a gamma-alumina layer. The laboratory analyses for composition were carried out by energy-dispersive X-ray mapping in a scanning electron microscope. The final aluminosilicate coating on the test insert consisted of powder flame-sprayed molybdenum 0.003 in. (0.076 mm) thick and γ-alumina + amorphous silica for a combined thickness of 0.003 in. (0.076 mm).

The plasma spray parameters for the aluminosilicate coating and the yttria-stabilized zirconia coating are presented in TABLE 2.

C. Procedure

The technique used in the experiments consists basically of the development and application of a technique suggested by Hurn, et al. (6) and Yu, et al. (7) As adapted and applied to the combustion bomb described previously, the technique consists of measuring the pressure change that occurs in the bomb during the injection process. These measurements are made under identical thermodynamic conditions, using the same spray characteristics with fuel being injected first into air and then into nitrogen. In a typical experiment, two pressure traces are obtained -- one with the
### TABLE 2. Plasma-Spray Parameters

<table>
<thead>
<tr>
<th>Powder</th>
<th>Molybdenum</th>
<th>Alumina</th>
<th>Silica</th>
<th>Amdry 995</th>
<th>Metco 204</th>
</tr>
</thead>
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<tr>
<td><strong>Spray Gun</strong></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type</td>
<td>3MB</td>
<td>3MB</td>
<td>3MB</td>
<td>3MB</td>
<td>3MB</td>
</tr>
<tr>
<td>Nozzle</td>
<td>GM</td>
<td>GM</td>
<td>GM</td>
<td>GA</td>
<td>G</td>
</tr>
<tr>
<td>Powder Port</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td><strong>Gas Pressure, psi</strong></td>
<td></td>
<td></td>
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<tr>
<td>Primary Nitrogen</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>55</td>
</tr>
<tr>
<td>Secondary Nitrogen</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
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<tr>
<td><strong>Power</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Amps</td>
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<td>500</td>
<td>400</td>
<td>500</td>
<td>500</td>
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<tr>
<td>Volts</td>
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<td>64-70</td>
<td>55</td>
<td>65</td>
<td>78-82</td>
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<tr>
<td><strong>Powder Feed</strong></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type</td>
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<td>3MP</td>
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<td>Gas Flow</td>
<td>37</td>
<td>37</td>
<td>41</td>
<td>37</td>
<td>37</td>
</tr>
<tr>
<td><strong>Spraying</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distance, in.</td>
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<td>2-4</td>
<td>2</td>
<td>3-4</td>
<td>4-5</td>
</tr>
<tr>
<td>Rate, g/hr</td>
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<td>25</td>
<td>45</td>
<td>45</td>
<td>45</td>
</tr>
</tbody>
</table>

Fuel injected into air with subsequent combustion, and one with the fuel injected into nitrogen. An example of the data obtained from the bomb is plotted in Fig. 2.

By comparing the pressure traces, it can be seen that, in both cases, the pressure decreases during the early part of the injection event. In the case with air, the pressure ultimately rises due to the onset of self-sustaining combustion. In the nitrogen case, the pressure remains below the baseline. In both cases, the pressure drop is due to the vaporization of the fuel. The physical delay ($T_p$) has in the past (8, 9)
been defined as the time measured from the start of injection to the point where nitrogen and air cases diverge. The chemical delay ($T_c$) is then defined as the time elapsed from the divergence point to the point at which the air case crosses the baseline. These definitions are somewhat arbitrary in that chemical reactions could occur during the defined physical-delay period, while the definition of chemical delay implies that the reactions become self-sustaining only after the cooling effect of fuel vaporization is overcome.

An improved definition of the physical delay time is based on the realization that the fuel vaporization and mixing are coupled, and affect the chamber pressure during the same time interval. The improved definition for the physical delay, therefore, is measured in terms of the elapsed time from the start of injection (needle lift rise) to the minimum of the chamber pressure trace. The definitions of the various points on the scope trace are also shown in Fig. 2.

Figure 2. Pressure-time records and injection-nozzle needle-lift trace with identification of the various points of interest
The question of defining the ignition delay time usually results from the difficulty of identifying the start of combustion. Several possibilities include the start of pressure rise, the start of heat release, or the occurrence of a luminous flame. In this work, the total ignition delay time \( T_d \) is defined as the time from the start of injection, as indicated by needle lift, to the initial pressure cross-point (see Fig. 2). It was felt that this definition was acceptable for these comparisons because of the similarity in the shapes of the combustion pressure traces.

The three inserts were tested in the CVCA at initial test conditions of 800°F (427°C) and 300 psia (2.0 MPa) using a 36 cetane number blend of the primary reference fuels for cetane rating. The experiments were performed starting with the fresh surface and repeating 24 times, recording the ignition delay time for each test. The uncoated insert was tested twice, once at the start of the experiments, and again after the other two coated inserts were evaluated.

IV. RESULTS AND DISCUSSION

The results of the experiments are tabulated in TABLE 3. A cursory examination of the data reveals that the ignition delay times are very similar for all the experiments. The mean for the entire data set is 17.0, with a standard deviation of 1.17. Measuring this effect on ignition in terms of cetane number, this represents a 1.77 cetane number difference, using the same fuel and tests condition. The extremes of the data set are 14.5 and 21.1 ms. This difference, which could actually include outliers of the data set, represents only a 5.0 cetane number difference. It can be concluded from these observations that the effects of the ceramic surfaces are small. In order to be able to detect any possible differences, it was felt that the data should be analyzed using statistical techniques designed to identify small differences in data sets.

The experimental data were entered into the computer and analyzed statistically to determine if there were any differences that could be related to the ceramic coating. The results of the analysis indicated that the two repeats of the baseline data were not significantly different, demonstrating that the results were not varying with time. In addition, there was no apparent relation between the ignition delay and completion of the repeats on a given insert, indicating that repeated combustions were not affecting the results. The results using the aluminosilicate (G-Al) coating were not significantly different, in a statistical sense, from the baseline. In addition, the results using the
TABLE 3. Tabulation of Experimentally Determined Ignition Delay Times

<table>
<thead>
<tr>
<th>Repeat No.</th>
<th>Baseline Before Experiment 1</th>
<th>Baseline Before Experiment 3</th>
<th>Baseline After Experiment 2</th>
<th>Baseline After Experiment 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>16.2</td>
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<tr>
<td>2</td>
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<td>3</td>
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<td>4</td>
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<td>17.1</td>
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<td>16.9</td>
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<td>14</td>
<td>16.6</td>
<td>18.0</td>
<td>18.2</td>
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<td>16.5</td>
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<td>18.7</td>
<td>15.2</td>
<td>16.1</td>
</tr>
<tr>
<td>24</td>
<td>17.2</td>
<td>18.0</td>
<td>17.4</td>
<td>16.3</td>
</tr>
</tbody>
</table>

The means and the standard deviations of the data for each of the different inserts are listed in TABLE 4. These results are plotted in Fig. 3 using the appropriate error band for each set of experiments. The statistical analyses provide a strict interpretation of the results, in which there are no statistically significant differences in the results for the coated surfaces as compared to the composite of the baselines. The plot, on the other hand, offers an opportunity to examine the data in a more qualitative sense, possibly providing a better insight into the true effects on ignition.
Table 4. Means and Standard Deviations for Each Insert

<table>
<thead>
<tr>
<th>Insert</th>
<th>Mean, ms</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline Before</td>
<td>17.3</td>
<td>1.24</td>
</tr>
<tr>
<td>PSZ</td>
<td>17.8</td>
<td>1.00</td>
</tr>
<tr>
<td>G-Al</td>
<td>16.4</td>
<td>0.98</td>
</tr>
<tr>
<td>Baseline After</td>
<td>16.6</td>
<td>0.91</td>
</tr>
</tbody>
</table>

Examination of Fig. 3 reveals that while "Baseline Before" and "Baseline After" overlap, the overlap is small and that the "Baseline After" appears to have a lower ignition delay time. The order of performing the experiments was "Baseline Before", insert G-Al, insert PSZ, and "Baseline After." If the results for the coated inserts are compared to
the baseline data generated in the closest corresponding experiments, the results for insert G-AI should be compared to "Baseline Before", and the results for insert PSZ should be compared to "Baseline After". If these comparisons are made, it becomes apparent that the results for insert G-AI are statistically lower than the "baseline before", while those for insert PSZ are statistically higher than the "baseline after". Based on the means of the four data sets, the partially stabilized zirconia (PSZ) produced a 0.6 increase in cetane number, while the aluminosilicate (G-AI) produced a 0.9 decrease in cetane number. It should be noted, however, that these effects are approximate and may not be significant in an engine during cold start when the surfaces are cold and the catalytic effects, if any, are probably even smaller.

As indicated previously, the potentials for fouling of the surfaces appear to be extremely high in an engine in which the surfaces are exposed not only to the fuel and combustion products but also to the lube oil. If the ceramic surfaces are to serve dual purposes of providing insulation as well as a catalytic surface, it can be argued the surface temperatures will be high and the reaction rates sufficient to prevent surface fouling. Indeed, the Morotsky patent claims this as a beneficial effect of the catalytic surface. Fig. 4 is a photograph of the inserts showing the before condition (small pieces at the top

![Figure 4. Inserts showing before conditions and condition after the experiments (small pieces at top of each insert show before condition)](TWR3.A)
of each fuel insert) and the condition after the experiments. It should be noted that the surfaces were maintained at 427°C (800°F), well above cylinder wall surface temperatures attainable in oil-lubricated diesel engines. It should also be noted that the coated inserts were exposed to only 24 combustion events. In addition to the fact that the surfaces had significant deposits, it is interesting that the alumina silicate surface appeared to have the least amount of deposit.

V. SUMMARY

In a strict statistical sense, the results of these experiments indicate that the ceramic coatings have no effect on the ignition delay time as determined in the CVCA. If, on the other hand, the drift in the experimental results are accounted for by using the baseline data developed nearest in time to the corresponding data for the coated inserts, it appears that the ignition process can be affected by the surface of the combustion chamber. In the case of the PSZ, the result was toward increased ignition delay time, an undesirable result for reduced cetane requirement in an actual engine. The fact that an effect is possible suggests that the PSZ should be studied in a more basic research program to determine if the results are physically induced or a result of chemical reaction. In addition, more coating should be studied to determine if positive effects can be obtained if the appropriate materials are selected and applied. These tests should include different applications of the aluminosilicate since the results were in the direction of reduced ignition delay for the application tested.
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