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Development of Wear Resistant
Ceramic Coatings for Diesel
Engine Components

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DEVELOPMENT OF WEAR-RESISTANT CERAMIC COATINGS FOR DIESEL ENGINE COMPONENTS

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ABSTRACT

1.0 Abstract

Improved fuel economy and a reduction of emissions can be achieved by insulation of the combustion chamber components to reduce heat rejection. However, insulating the combustion chamber components will also increase the operating temperature of the piston ring/cylinder liner interface from approximately 150 C to over 300 C. Existing ring/liner materials can not withstand these higher operating temperatures and for this reason, new materials need to be developed for this critical tribological interface. The overall goal of this program is the development of piston ring/cylinder liner material pairs which would be able to provide the required friction and wear properties at these more severe operating conditions. More specifically, this program first selected, and then evaluated, potential wear resistant coatings which could be applied to either piston rings and/or cylinder liners and provide, at 350 C under lubricated conditions, coefficients of friction below 0.1 and wear rates of less than 25×10^{-6} mm/hour.

The processes selected for applying the candidate wear resistant coatings to piston rings and/or cylinder liners were plasma spraying, chemical vapor, physical vapor and low temperature arc vapor deposition techniques as well as enameling techniques. The adherence of each coating and application process selected to either cast iron (for potential cylinder liner coatings), H-13 tool steel or 17-4 PH stainless steel (for potential piston ring coatings) was determined using a modified tensile pull adherence test. Except for two ceramic coating candidates, all the coatings had acceptable adherence. The two plasma sprayed ceramic coatings which had unacceptable adherence had significantly lower thermal expansion coefficients than the metallic substrates on which they were applied. A graded coating, containing various layers containing different ratios of metallic bond coat to ceramic, was devised for these coatings to gradually reduce these differences in thermal expansion. These graded coatings had significantly improved adherence.

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Next, each coating was screened for friction and wear at 350 C, lubricated, using a Falex Pin-on-Disk friction and wear test machine. Using the results of the pin-on-disk screening as a guide, 12 potential piston ring and cylinder liner coating pairs were selected. These coating pairs were then further optimized for friction and wear using a Hohman A-6 Double Rub Shoe friction and wear machine. Again, the testing was done at 350 C, under lubricated conditions.

Upon completion of the Hohman A-6 optimization, the following two piston ring/cylinder liner coating pairs were identified as meeting the friction and wear goals of this program:

1. A plasma sprayed high carbon iron-molybdenum piston ring coating running against a plasma sprayed chromia-silica cylinder liner coating; and,
2. A plasma sprayed high carbon iron-molybdenum piston ring coating running against a low temperature arc vapor deposited chrome nitride coating.

The thermal shock (down-cycle from 650 C to 100 C) and oxidation resistance (500 hours at 400 C in a simulated diesel engine exhaust atmosphere) of these coatings were determined next. In addition, a complete wear curve for each coating pair was obtained. All the coatings were able to withstand the thermal shock and oxidation testing without any coating spallation or excessive oxidation. The wear curves showed that the high carbon iron-molybdenum and chromia-silica coatings, if applied at thicknesses above 15 microns, will meet commercial diesel engine durability requirements. The chrome nitride coating also will meet the commercial diesel engine durability requirements if it is applied at thicknesses above 15 microns. However, current LTAVD chrome nitride coating technology can only deposit 3-5 micron thick chrome nitride coatings. Therefore, coating techniques need to be developed and optimized for applying adherent, 15 micron thick, chrome nitride coatings to cast iron substrates.

Finally, simulated piston ring and cylinder liner specimens were coated and sent to Battelle for additional friction and wear characterization in their high temperature friction and wear test rig.

2.0 Introduction

2.1 Background

The development of low heat rejection diesel engine technology involves insulating the combustion chamber components to achieve improvements in both fuel economy and emissions. Insulating the combustion chamber components increases the cylinder operat

ing temperatures above those currently found in existing engine designs. Materials used in current diesel engine designs will not meet the commercial durability requirements if the cylinder temperatures are increased much over the current temperatures. New materials, which can meet these durability requirements, need to be developed so that the advantages of insulating the engine to reduce heat rejection can be achieved.

One of the critical tribological interfaces which needs to be improved, due to the implementation of insulated components, is the piston ring/cylinder liner interface. These interfaces in current diesel engine designs operate at approximately 150 C and can preform in excess of 10,000 hours of engine operation using conventional diesel fuels before they need to be replaced. Insulating the combustion chamber can increase the piston ring/cylinder liner interface temperature to 300 C or above. Current piston ring and cylinder liner materials can not withstand these operating conditions. For this reason, wear coatings, which can be applied to piston rings and cylinder liners, need to be developed for these advanced low heat rejection diesel designs.

The overall objective of this program is the development of adherent, wear resistant coatings which can be applied to either piston rings and/or cylinder liners. These advanced wear coatings must have a lubricated friction coefficient of 0.1 or less, and a wear rate of less than 25×10^{-6} mm/hour with the following test conditions: boundary lubrication, a pin load of 2.3 kilograms, and a velocity of 206 meters per second. The wear goal of 25×10^{-6} mm/hour is consistent with the need to have the piston rings and cylinder liners which can operate for 10,000 hours or 500,000 miles before replacement.

2.2 Program Plan

Figure 2.2.1 contains the overall program plan. The program began by first selecting potential coating application processes and wear resistant coating compositions which can be applied to piston ring and/or cylinder liners. Selection criteria for each coating and application method were published mechanical and physical property data as well as any friction and wear property data available in the open literature. Also considered in this selection process, were the ease and cost of applying each coating to either an outer diameter of a piston ring or the inner diameter of a cylinder liner, and any post treatment required (i.e., diamond grinding).

After completion of the coating selection process, the potential wear resistant coatings were applied to various the metallic substrates and the adherence of each coating to the substrate was determined. If required, compositional or application process changes were made to

improve the adherence of the wear resistant coatings to the metallic substrate. Then using the application process, adherence, and physical appearance as selection criteria, potential piston ring and cylinder liner wear resistant coatings were selected for friction and wear screening. This screening was performed on a Falex pin-on-disk friction and wear tester at 350 C, lubricated, running against both ceramic and metallic counterfaces.

Figure 2.2.1 Program Plan

The pin-on-disk friction and wear screening identified 12 wear resistant piston ring and cylinder liner coating pairs which had the potential for meeting the friction and wear goals of this program. The friction and wear properties of these coating pairs were next optimized using an Hohman A-6 Double rub shoe friction and wear test machine. Again, the friction and wear testing was performed at 350 C under lubricated conditions. Upon completion of this friction and wear optimization, two piston ring/cylinder liner coating pairs were identified as meeting the friction and wear goals of this program. Finally, these coating pairs were fully characterized with respect to oxidation resistance, thermal shock resistance, coating uniformity, adherence to the metallic substrate, and friction and wear.

2.3 Program Approach

A systems approach was selected for meeting the program objectives. This approach was selected because of the interdependence of the variables which can affect the friction and wear properties of a coating. Two important variables which can have a significant effect on the friction and wear properties of a coating are the compatibility of the coating with the lubricant and the surface it is sliding against.

The compatibility of the coating with the lubricant is very important. Incompatibilities of the coating with the lubricant can lead to coating corrosion, lubrication degradation, or a malfunction of the lubricating film. All these effects can significantly reduce the life of the tribological system. Compatibility of the sliding pairs is important for reducing potential problems such as catastrophic abrasive or adhesive wear and/or galling.

For these reasons, the best wear resistant piston ring and cylinder liner coating pairs were selected as early in the program as possible. In addition, only one high temperature lubricant was used throughout the program. This lubricant was used for both the initial pin-on-disk friction and wear screening as well as the subsequent Hohman A-6 friction and wear optimization.

3.0 Technical Discussion

3.1 Selection of Coating Compositions and Application Processes

There are many coating deposition techniques which can apply adherent, wear resistant coatings to both the outer diameter of piston rings and the inner diameters of cylinder liners. These coating depositions techniques can be separated into four distinct groups: atomistic deposition, particulate deposition, surface modification and bulk coating. Table 3.1.1 lists the four general coating deposition techniques and the various specific coating processes included under each general coating technique. For example, chemical and physical vapor deposition is listed under atomistic deposition while plasma spraying is listed under particulate deposition.

Table 3.1.1 Four General Coating Deposition Techniques

Atomistic Deposition	Particulate Deposition
<ul style="list-style-type: none"> - Electrolytic Environment - Vacuum Environment - Plasma Environment - Chemical Vapor Environment - Liquid Phase Epitaxy 	<ul style="list-style-type: none"> - Thermal Spraying - Fusion Coatings - Impact Plating
Bulk Coatings	Surface Modification
<ul style="list-style-type: none"> - Wetting Processes - Electrostatic Spraying - Cladding - Overlaying 	<ul style="list-style-type: none"> - Chemical Conversion - Chemical-Liquid - Chemical-Vapor - Leaching - Mechanical - Thermal Surface Enrichment - Sputtering - Ion Implantation

Selection of the coating deposition techniques to be evaluated was made by using preliminary base manufacturing and cost considerations. The variables included in the preliminary base manufacturing cost were the ability of the process to easily coat the desired surface, production experience, if any, with coating deposition technique, potential for automation of the coating process, substrate surface preparation required prior to coating application, number of coating steps required to obtain the desired coating thickness, processing time, tolerance to processing variations, and the finishing requirements after coating application.

The preliminary base manufacturing and cost considerations identified five coating deposition techniques. The five coating techniques identified were: plasma spraying, chemical vapor deposition (CVD), physical vapor deposition (PVD), low temperature arc vapor deposition (LTAVD), and enameling.

Plasma spraying was selected because it is a current production process for the application of wear resistant coatings to piston rings. Caterpillar uses plasma sprayed chrome carbide piston rings in several types of production engines. An advantage of plasma spraying is that there are many powder compositions commercially available which can be applied to a wide range of metallic substrates with varying chemistries. Also, because the starting materials for plasma spraying are powders, they can be easily tailored to fit an application. For example, solid lubricants can be blended into the plasma spray powder to reduce the coefficient of friction of the applied coating, or hard particles, such as chrome carbide can be added to the powder composition to reduce the wear coefficient. Finally, powders of different chemistries can be blended together to make the thermal expansion coefficient of the plasma coating match the thermal expansion coefficient of the metallic substrate.

Plasma spray systems are available for coating both the outer diameter of piston rings (O.D. gun) and the inner diameter of cylinder liners.

After a reviewing the available literature, the following plasma spray coatings compositions were selected for evaluation as potential wear resistant coatings for either piston ring or cylinder liner applications: a chromia-silica composite; a high carbon iron-molybdenum blend; a crystalline chrome carbide, a nickel-chromium alloy; a high cobalt, molybdenum-chromium alloy; NASA's self-lubricating PS 212, a high molybdenum-nickel-chromium alloy; zirconia; and a zirconia-titania-yttria composite. The plasma spray coating compositions selected included hard-facing compositions and self-lubricating compositions.

Chemical and/or physical vapor deposited coatings are widely used on cutting tools for extended tool life. Titanium nitride and titanium carbide CVD/PVD coatings are examples of commercial coatings. In addition, preliminary work at Caterpillar has shown that applying a PVD titanium nitride coating to a piston ring can improve the wear characteristics of the ring.

An advantage of using CVD, PVD or low temperature arc vapor deposited (LTAVD) coatings is that they conform to the surface to which they are applied. This means that if they are applied to a surface with a 5 to 10 micro-inch surface finish, the CVD, PVD or LTAVD coatings will also have a 5 to 10 micro-inch surface finish. Thus, these coatings will not require any costly machining operation after application.

Three different vapor deposited coating processes were selected because each process offers certain advantages. The PVD or LTVAD coatings are applied at lower temperatures (300-500 C) than a CVD coating. The 1000 C processing temperature, required for CVD processing, usually requires that the metallic substrate be reheat treated after CVD coating to obtain the desired surface hardness required for acceptable wear resistance. With both the PVD or LTVAD, since the coating is applied at lower temperatures, a post heat treatment after coating is not required.

A mid-temperature CVD process has been developed for application of titanium carbonitride coatings to a 17-4 PH substrate. The processing temperature for this coating is 800 C and during the cool-down the 17-4 can be heat treated to obtain the proper hardness. The 200 C lower application temperature for this process also will reduce the amount of distortion which occurs to piston rings during coating with a conventional CVD process. Again, piston ring distortion will not be a problem with either the PVD or LTAVD processes due to their lower application temperatures.

One advantage of the CVD process is it can be used for applying thicker coatings (10-15 microns) than either the PVD or LTAVD coating processes (3-5 microns). Unlike the lower temperature coating processes, a layered composite coating can be applied using the CVD process. Finally, the higher processing temperatures for the CVD process, the CVD coatings usually have better adhesion to the metallic substrate than the PVD or LTAVD coatings.

Both the CVD and PVD are potential techniques for applying wear resistant coatings to piston rings. These techniques, however, are not suited for coating the inner diameter of cylinder liners. The LTAVD process, on the other hand, uses a directed source. Thus, this technique can be used to apply wear resistant coatings to the inner diameters of surface finished cylinder liners.

Chemical, physical, and low temperature arc vapor deposited coating processes were selected. The chemical and physical vapor deposited coating techniques were used to apply wear resistant coatings to piston rings. The low temperature arc vapor deposition technique was evaluated as a potential technique for applying a wear resistant coatings to the inner diameters of cylinder liners. The following coatings from each process were chosen for evaluation:

1. Chemical Vapor Deposited Coatings — chrome carbide, a layered titanium carbide, titanium nitride, and a tungsten/ tungsten carbide mixture,
2. Mid-temperature Chemical Vapor Deposited Coating — Titanium carbonitride,
3. Physical Vapor Deposited Coating — Titanium nitride, and
4. Low Temperature Arc Vapor Deposited Coating — Chrome nitride.

Enameling was the third application process selected. Enameling offers several cost advantages over plasma spraying or chemical, physical or low temperature arc vapor coating techniques. The enamel coatings use inexpensive raw materials and can be applied to the inner diameters of cylinder liners using conventional robotic slurry spraying techniques. In addition, the enamel compositions can be tailored to adhere strongly to and closely match the thermal expansion coefficient of cast iron substrate. Most importantly, many enamels after application and firing, have very smooth, hard surfaces. These surfaces may require only a minimum amount of machining to obtain the proper surface finish.

Two enameling processes were evaluated for producing wear resistant enamel coatings. The first uses the vitrious phase coating (VPC) technology developed at Solar Turbines, Inc. This technology was developed for applying oxidation resistant coatings to gas turbine components. The VPC technology uses water to suspend the enamel frit, slurry spray techniques for applying the frit to the metallic substrate, and a firing cycle for drying and fusing the enamel coating to the metallic substrate. Hard particles such as chrome oxide, alumina or zirconia can be added to the vitrious phase composition to improve the wear resistance of the enamel. In summary, the VPC approach uses an enamel composition as the adhesive for bonding hard particles to a metallic substrate.

The second enameling approach was developed by the University of Illinois. An enameling technique called the "hot substrate method," produced a wear resistant enamel coating by sprinkling and/or plasma spraying hard particles on top of a molten enamel layer. The wear resistant enamel coating produced by this method, consists of hard particles embedded within the enamel.

The enamel compositions selected for both techniques had thermal expansion coefficients which closely matched that of the cast iron substrate. The reason for the matching of the thermal expansion coefficients was to reduce the amount of residual stresses which develop

within the enamel and/or metallic substrate either during the cool-down after the initial firing cycle or during the combustion cycle heat-up to 350 C. The residual stresses which develop due to thermal expansion differences could cause the enamel coating to disbond or spall from the metallic surface.

3.2 Optimization of Plasma Sprayed, Chemical and/or Physical Deposited, and Enamel Candidates for Coating Adherence

3.2.1 Plasma Coatings

3.2.1.1 Thermal Expansion Determinations

The thermal expansion coefficient of a plasma coating applied to a metallic substrate should closely match the thermal expansion coefficient of the substrate. Large differences in thermal expansion coefficients can result in the development of residual stresses, either in the coating or substrate. These residual stresses can lead to coating failure.

The thermal expansion coefficient of each candidate plasma spray coating was determined from room temperature to 1000 C. All the thermal expansion determinations were made using a Theta Industries, Inc., High Temperature Double Pushrod Dilatometer. The specimens were hollow cylinders of each of the plasma sprayed powders. These specimens were obtained by plasma spraying each coating composition on a metal rod, cutting the specimens to length, and finally, removing the metal rod by acid etching.

The thermal expansion coefficients were determined for both the graded and nongraded plasma sprayed coatings as well as the cast iron, H-13 tool steel, and 17-4 PH stainless steel substrates. The results are listed in Table 3.2.1. The grey cast iron and H-13 tool steel had thermal expansion coefficients at 400 C of 12.2×10^{-6} C and 14.4×10^{-6} C, respectively. The thermal expansion coefficients of these metallic substrates are very similar to the thermal expansion coefficients of the following plasma spray coatings: NiCrAlY bond coat; nickel-chromium alloy; high cobalt, molybdenum, chromium alloy; high carbon iron-molybdenum blend; NASA self-lubricating PS 212; and high molybdenum, nickel, chromium alloy. As a result, these plasma spray coatings can be applied to either the cast iron or H-13 tool steel and can be heated to 400 C without creating large residual stresses in either the plasma coating or metallic substrate. The zirconia, and titania, yttria composite plasma coatings, on the other hand, had thermal expansion coefficients significantly lower than those of the metallic substrate. These coatings, if applied to either the cast iron or H-13 substrate, could spall or disbond from the metallic substrate either after application or when the component is heated to 400 C if a method for minimizing the thermal expansion mismatch stresses is not used.

Table 3.2.1 Thermal Expansion Coefficients (10^{-6} C)

SUBSTRATES	200 C	400 C
H-13 Tool Steel	11.5	12.2
Gray Cast Iron	12.3	14.4
PLASMA SPRAY COATINGS		
Nickel-Chrome Alloy	10.7	11.7
High Cobalt-Molybdenum Alloy	9.9	11.3
Chrome Carbide with Solid Lubricants	9.6	11.3
High Molybdenum, Nickel, Chrome Alloy	10.6	11.3
High Carbon Iron-Molybdenum Alloy	8.8	11.1
Zirconia, Titania, Yttria Powder	7.6	8.4
Zirconia	8.0	8.0
Molybdenum	4.2	4.8
Bond Coat	12.3	13.7

The effect of grading plasma coatings on thermal expansion was determined by Caterpillar in contract DEN3-332 of the DOE/NASA Heavy Duty Transport Technology program. In this program, it was learned that the thermal expansion coefficients of plasma spray coatings can be tailored to match the thermal expansion coefficient of a metallic substrate by blending plasma coatings with significantly different thermal expansion coefficients. The thermal expansion coefficient of this coating will follow the rule of mixtures. That is, a 50/50 blend, by volume, will have a thermal expansion coefficient midway between that of the individual plasma spray coatings.

Table 3.2.2 illustrates the effect of blending the zirconia plasma spray coating with the bond coat on the thermal expansion coefficient. Using different zirconia and bond coat ratios, a graded plasma coating system was developed for both these ceramic coatings. The graded coating systems had graded thermal expansion coefficients—matching the metallic substrate on one end, and the zirconia top coating on the other end.

Table 3.2.2 Effect of Grading on Thermal Expansion Coefficients (10^{-6} C)

Substates	400 C
H-13 Tool Steel	12.2
Gray Cast Iron	14.4
Graded Plasma Coating Compositions	
100% Zirconia	8.1
75% Zirconia/ 25% Bond Coat	9.5
50% Zirconia/ 50% Bond Coat	10.9
25% Zirconia/ 75% Bond Coat	11.0
100% Bond Coat	13.7

(All percentages are volume percent)

3.2.1.2 Plasma Spray Coating Adherence Determinations

Using thermal expansion determinations as a guide, the adherence of the following plasma spray coatings was determined, after application to either cast iron and/or H-13 tool steel substrates, without grading: nickel-chromium alloy; high cobalt, molybdenum, chromium alloy; chromia-silica composite; high carbon iron-molybdenum blend; high molybdenum, nickel, chromium alloy; and NASA self-lubricating PS212. The thermal expansion determinations also showed that graded coating schedules had to be developed for the zirconia and zirconia-titania-yttria coatings. The adherence of these graded coatings was also determined.

The adherence determinations were made using the tensile pull adherence test illustrated in Figure 3.2.1. In general, the test setup consisted of two plasma sprayed specimens bonded to two uncoated substrates. The epoxy selected for bonding the components together had a shear strength of 27.6 MPa. After curing the epoxy, the two uncoated substrates were pulled in tension. Pulling the uncoated specimens in tension caused a shear stress to develop within both the plasma sprayed coating and the epoxy. In this test, if the shear strength of the plasma coating or plasma coating/ metallic interface was greater than the shear strength of the epoxy, the specimen failed at a load of 27.6 MPa. When the shear strength of either the plasma spray coating or the coating/ metallic interface was less than the shear strength of the epoxy, the specimen failed at loads below 27.6 MPa, and the failure occurred within the coating or at the coating/ metallic interface.

The adherence of all the candidate plasma coatings was determined. These results are listed in Table 3.2.3. The following plasma coatings had adherence values >27.6 MPa when applied to either the cast iron or H-13 tool steel substrates: high cobalt, molybdenum, chromium alloy; high carbon iron-molybdenum blend; high molybdenum, nickel, chromium alloy; high molybdenum composite; and the NASA PS212. The nickel-chromium alloy recorded a 17.4 MPa adherence when applied to the cast iron substrate. This coating spalled after application when plasma sprayed onto the H-13 tool steel. Finally, the chromia-silica composite recorded 13.2 and 9.7 MPa, adherence values for the cast iron and H-13 tool steel, respectively. It should be noted that, since these coatings did not spall during the initial pin-on-disk friction and wear screening, adherence values above 9.7 MPa can be considered acceptable tensile pull adherence values.

The adherence determinations for the graded coatings are listed in Table 3.2.4. These results show that for the coatings applied to the cast iron, grading reduced the adhesion of the coating to the substrate. However, because all the adherence values were above 9.7 MPa, this reduction in adhesion should not be large enough to cause coating spallation in a sliding wear application. The adhesion of the graded zirconia coatings was poorer on the

H-13 tool steel than the cast iron. Also, grading had little effect on the adhesion. Finally, grading the zirconia, titania, yttria composite improved the adhesion of this coating when it was applied to an H-13 tool steel substrate.

Table 3.2.3. Tensile Pull Adherence Results--Plasma Sprayed Coatings

COATING	ADHERENCE RESULTS	
	GRAY CAST IRON	H-13 TOOL STEEL
Nickel, Chrome Alloy	17.4 MPa	Spalled
High Cobalt, Molybdenum, Chrome Alloy	>27.6 MPa	>27.6 MPa
Chromia-silica Composite	13.2 MPa	9.7 MPa
High Carbon Iron-Molybdenum Composite	>27.6 MPa	>27.9 MPa
High Molybdenum, Nickel, Chrome Alloy	>27.6 MPa	>27.6 MPa
High Molybdenum Composite	>27.6 MPa	>27.6 MPa
PS 212	>27.6 MPa	>27.6 MPa

Table 3.2.4. Tensile Pull Adherence Test Results--Graded Coatings

Graded Coating Composition	Adherence Results	
	Gray Cast Iron	H-13 Tool Steel
Zirconia Top Coat/ Bond Coat	20.1 MPa	11.2 MPa
Zirconia Top Coat/ 50-50/ Bond Coat	15.0 MPa	13.1 MPa
Zirconia Top Coat/ 50-50/ 25-75/ Bond Coat	13.3 MPa	11.9 MPa
Zirconia-Titania-Yttria/ Bond Coat	>27.6 MPa	12.9 MPa
Zirconia-Titania-Yttria/ 50-50/ Bond Coat	10.8 MPa	20.3 MPa

FIGURE 3.2.1

SCHEMATIC OF THE TENSILE PULL TEST FOR MEASURING ADHERENCE

3.2.2 Chemical and/or Physical Deposited Coatings

3.2.2.1 Physical Appearance

The high temperature chemical vapor deposited (CVD) chrome carbide and layered titanium carbide/titanium nitride coatings were applied to H-13 tool steel substrates. The mid-temperature CVD titanium carbonitride applied to a 17-4 stainless steel substrate. These CVD coatings had an excellent physical appearance and no coating flaking or spalling was noted on any of the specimens. An optical microscopy analysis performed on these specimens indicated that the coatings were uniformly applied, and no adverse chemical reactions between the coatings and substrate occurred during the coating process. Using cross-sectional optical photomicrographs the thicknesses of the CVD coatings were determined to be:

Chrome carbide—5 microns,
Titanium carbonitride—14 microns,
Layer titanium carbide/titanium nitride—8 microns.

A physical vapor deposited (PVD) titanium nitride coating was also applied to the H-13 tool steel substrate. A metallographic analysis of this coating showed that the PVD titanium nitride coating was not uniform, varying in thickness from less than 1 micron to over 3 microns across the three inch width of the specimen.

The CVD tungsten/tungsten carbide coating applied to the H-13 tool steel substrate had an excellent physical appearance. However, the tungsten/ tungsten carbide coating cannot bond directly to the H-13 tool steel substrate. For this reason, a nickel oxide layer was first applied and the tungsten/ tungsten carbide coating was deposited on top of the nickel oxide layer. Problems were encountered obtaining a smooth, uniform nickel oxide layer on the H-13 tool steel substrate. As a result, the surface roughness of the tungsten/tungsten carbide coated specimens ranged from 10 to 50 micro-inches.

3.2.2.2 Adherence Determinations

At first, a Revetest was used for determining the mechanical strength; i.e., adhesion and intrinsic cohesion, of the chemical and physical vapor deposited coatings. In general, the Revetest scratches the surface of the coated substrate with a smoothly rounded diamond point while simultaneously applying a load to the tip. The load is increased until a piezoelectric accelerometer detects an acoustic emission as the coating is disturbed. Depending on the nature of the coating disturbance; i.e., cracking, flaking, smearing, the pattern and intensity of the acoustic emission varies.

Figures 3.2.2 and 3.2.3 contain the Revetest results obtained from two mid-temperature chemical vapor deposited titanium carbonitride coated specimens applied to H-13 tool steel substrates. These specimens were processed together in the same reactor. The Revetest results for these specimens show that for one specimen the maximum acoustic emission occurred at a 1000 gram load while for the second specimen the maximum acoustic emission occurred at a load of 200 grams. Also, the shape of the load versus maximum acoustic emission for each specimen was very different. Optical photomicrographs of with each Revetest trace illustrate that the physical appearance of the scratches were identical on both specimens. Thus, the results obtained from the Revetest on two specimens with identical coatings were very different. Finally, the comparison of the Revetest results obtained from different chemical vapor deposited coating compositions also provided inconclusive results. For this reason, it was decided to use the tensile pull adherence test for determining the adherence of all chemical and physical vapor deposited specimens.

**FIGURE 3.2.2
REVITEST RESULTS
SPECIMEN 1**

**FIGURE 3.2.3
REVITEST RESULTS
SPECIMEN 2**

All chemical and physical vapor deposited specimens failed at loads greater than 27.6 MPa. Examination of the specimens after testing showed that the failure occurred within the epoxy. Thus, all chemical and physical deposited coatings were determined to have acceptable adherence.

The physical vapor deposited titanium nitride coating was not included in the adherence determinations because the optical microscopy analysis of this coating showed it was non-uniformly applied to the H-13 tool steel substrate.

3.2.3 Enamel Coatings

The criteria for selecting an enamel composition involved an enamel composition which had a thermal expansion coefficient matched closely to the thermal expansion of the metallic substrate. In addition, the chemistry and firing cycle for the enamel must not cause an excessive amount of oxidation of the metallic substrate. Excess oxidation of the metallic substrate during firing can adversely affect the adherence of the enamel to the substrate. Finally, enamel compositions with low elastic moduli and favorable Poisson's ratios were selected. These properties are important when enamels are loaded with the hard particles.

The Solar VPC enamel composition required development of a bond coating to the metallic substrate prior to the VPC enamel. The bond coat had a similar chemistry as the VPC enamel but also contained NiO , Co_3O_4 , and MnO_4 . Without first applying the bond coat the adherence of the VPC enamel to the cast iron substrate was only marginal.

After selection of the enamel composition, it was loaded with 50 volume percent silicon carbide hard particles. During the firing of the enamel the silicon carbide reacted with the molten enamel producing very porous coatings. Thus, to prevent this undesirable chemical reaction from occurring, the hard particle additions should be oxides, such as chrome oxide, or stable carbides (i.e., chrome carbide).

The University of Illinois successfully plasma sprayed zirconia, chrome oxide, and chrome carbide over an A.I. Andrew's cobalt based enamel composition. These specimens had to be machined after plasma spraying to achieve the desired surface finish.

Sprinkling silicon carbide or silicon nitride onto a molten enamel resulted in very porous, rough coatings which were poorly bonded to the metallic substrate.

3.3 Pin-on-Disk Screening of Candidate Coatings for Wear and Friction (350 C, Lubricated)

3.3.1 Description of Pin-On-Disk Test Procedure

Screening of candidate coatings for friction and wear characteristics was performed on a Falex No.6 Multi-specimen Test Machine. Figure 3.3.1 illustrates the pin-on-disk test configuration. Tests were performed at 350 C with a normal load of 22.25 N and a surface velocity of 3.4/sec. The test temperature was attained by a combination of resistive heating coils embedded in the walls of the oil reservoir surrounding the test specimens and heating elements placed in cavities drilled into the lower table on which the disk specimen was mounted.

The contact surface of the test specimens was completely submerged in the test lubricant, an experimental synthetic lubricant supplied by Lubrizol. Oil was delivered to the reservoir by a peristaltic pump at a rate of 3.6 ml/min., allowing the oil to have approximately a three minute residence time in the reservoir. The candidate wear resistant coatings were applied to either cast iron or H-13 tool steel disks. Partially stabilized zirconia, AISI 440C stainless steel, alumina, or silicon nitride pins were used as the opposing counterfaces. Each test was run for 30 minutes before the test was stopped to measure the wear scar on the diameter of the pin as well as the coated disk. A Form Talysurf measured the extent of wear which occurred during each test. If the wear rate after the initial 30 minutes of testing was acceptable, the specimens were reassembled for further testing and the procedure repeated until a total of 2 hours of test time was accumulated.

FIGURE 3.3.1 SCHEMATIC OF THE PIN-ON-DISK

FRICTION & WEAR TEST

3.3.2 Wear Resistant Plasma Spray Coatings

The thermal expansion and adherence determinations identified the following six plasma spray coatings as potential wear resistant piston ring coatings: chromia-silica composite; high carbon iron-molybdenum alloy; NASA self-lubricating PS 212; high cobalt-chromium alloy with TRIBALLOY intermetallic additions; high molybdenum, nickel, chromium blend; and nickel, chromium, molybdenum blend. In addition, the thermal expansion and adherence determinations identified the following four coatings as potential wear

resistant cylinder liner coatings: chromia-silica composite; high carbon iron-molybdenum alloy; zirconia, titania, yttria blend; and the zirconia graded with NiCrAl bond coat. Tables 3.3.1 and 3.3.2 contain summaries of the pin-on-disk friction and wear screening results for these plasma sprayed coatings. The individual test data is contained in Tables 3.3.3 to 3.3.6.

Running either a ceramic or metallic counterface against the chromia-silica composite produced low coefficient of friction values (0.09-0.13) and average wear coefficients of 10^{-7} mm³/N-m for the coating and 10^{-8} mm³/N-m for either counterface. Optical photomicrographs of the wear track for this coating are contained in Figure 3.3.1. These photomicrographs show that the wear mechanism, which occurred during the test, was the ceramic counterface polishing the surface of the chromia-silica.

The high carbon iron-molybdenum alloy also recorded coefficients of friction between 0.09-0.13 running against both the ceramic and metallic counterfaces. This plasma coating exhibited average wear coefficients of 10^{-8} and 10^{-9} mm³/N-m, respectively, running against the ceramic and metallic counterfaces.

Coefficient of friction values (0.04-0.13) were obtained running the PS 212 coating against either the ceramic or metallic counterfaces. However, the average wear coefficients of 10^{-6} to 10^{-7} mm³/N-m obtained running PS212 against the ceramic and metallic counterfaces were borderline.

Both the high carbon-chromium alloy with TRIBALLOY intermetallic materials and the high molybdenum, nickel, chromium coatings exhibited high coefficient of friction values (0.18-0.33) running against either counterface. Average wear coefficients obtained from these coatings ranged between 10^{-8} and 10^{-7} mm³/N-m running against the ceramic counterface. Average wear coefficient of 10^{-6} mm³/N-m was obtained running a metallic counterface against the high molybdenum, nickel, chromium coating. For this reason, a 2.5 mm wear scar developed in the coating after only 60 minutes of testing. The photomicrographs in Figure 3.2.2 show that the wear mechanism for the high molybdenum, nickel, chromium coating was abrasive wear. The reason for the poor wear properties was the cracking and subsequent spalling of this coating under the load of the ceramic counterface.

The nickel, chromium, molybdenum plasma coating recorded low coefficient of friction values (0.07-0.13) as well as low average coating and counterface wear coefficients (10^{-8} mm³/N-m) running against the ceramic counterface. Higher coefficient of friction values (0.15-0.26) and average wear coefficients (10^{-7} to 10^{-6} mm³/N-m) were observed running this coating against a metallic counterface.

Table 3.3.1
Summary of Pin-On-Disk Friction and Wear Testing Candidate
Piston Ring Coatings Running Against the Zirconia Counterface

Plasma Sprayed Coatings	Friction		Average Wear Coefficient (mm ³ /N-m)	
	Min.	Max.	Disk Coating	Pin
Chromia-silica	0.08	0.15	4.69x10 ⁻⁷	1.19x10 ⁻⁸
High Carbon Iron-Molybdenum	0.05	0.15	1.50x10 ⁻⁷	7.50x10 ⁻⁹
PS 212	0.07	0.14	2.00x10 ⁻⁷	4.71x10 ⁻⁸
High Cobalt-Chromium with Tribaloy Intermed.	0.18	0.28	1.49x10 ⁻⁶	3.32x10 ⁻⁷
Nickel, Chromium, Molybdenum	0.08	0.13	3.72x10 ⁻⁸	1.98x10 ⁻⁸
High Molybdenum, Nickel, Chromium	0.08	0.21	5.7x10 ⁻⁸	3.28x10 ⁻⁷

- Chromia-silica, High carbon iron-molybdenum, and PS 212 all will be evaluated further because they met the program goals with respect to both friction coefficient and pin and disk wear rates.

- High Cobalt-Chromium with Tribaloy Intermediate and High Molybdenum, Nickel, Chromium coatings were dropped because of high coefficient of friction and either pin or disk wear coefficients.

CVD/PVD Coatings	Friction		Average Wear Coefficient (mm ³ /N-m)	
	Min.	Max.	Disk Coating	Pin
Mid-temperature Ti(C,N)	0.08	0.10	4.01x10 ⁻⁸	4.25x10 ⁻⁸
CVD Chrome Carbide	0.09*	0.18	4.73x10 ⁻⁷	4.25x10 ⁻⁸
CVD Layered TiC/TiN	0.10	0.16	8.80x10 ⁻⁸	7.60x10 ⁻⁹

* Test ran only for 60 minutes

- CVD Chrome Carbide coating was dropped because of a high disk wear coefficient.

- CVD layer TiC/TiN coating was dropped because although it had similar friction and wear results as the Mid-temperature Ti(C,N), it's processing was more complex and costly.

FIGURE 3.3.1
SURFACES AFTER PIN-ON-DISK TESTING
PLASMA SPRAYED CHROMIA-SILICA COMPOSITE

MAGNIFICATION 50X

MAGNIFICATION 200X

Table 3.3.2
Summary of Pin-On-Disk Friction and Wear Testing Candidate
Cylinder Liner Coatings Running Against the Zirconia Counterface

Plasma Sprayed Coatings	Friction		Average Wear Coefficient (mm ³ /N-m)	
	Min.	Max.	Disk Coating	Pin
Chromia-silica	0.08	0.15	4.69x10 ⁻⁷	1.19x10 ⁻⁸
High Carbon Iron-Molybdenum	0.05	0.15	1.50x10 ⁻⁷	7.50x10 ⁻⁹
LTAVD Chrome Nitride	0.09	0.19	3.10x10 ⁻⁸	2.84x10 ⁻⁸
Graded Zirconia	0.14	0.19	1.86x10 ⁻⁶	1.23x10 ⁻⁷
Zirconia, Titania, Yttria Composite	0.09	0.19	3.10x10 ⁻⁸	2.04x10 ⁻⁸

- Both the Graded Zirconia and Zirconia, Titania, Yttria composite were dropped because of high coefficient of friction and wear coefficients

Enamel Coatings	Friction		Average Wear Coefficient (mm ³ /N-m)	
	Min.	Max.	Disk Coating	Pin
Base Enamel (BE)	0.23	0.41	5.94x10 ⁻⁵	1.18x10 ⁻⁶
BE + 50 vol.% Alumium Oxide	0.18	0.18	6.48x10 ⁻⁷	4.71x10 ⁻⁶
BE + 50 vol.% Chrome Oxide	0.42	0.38	3.00x10 ⁻⁴	6.86x10 ⁻⁶
BE + 50 vol.% Zirconium Oxide	0.22	0.38	8.33x10 ⁻⁵	1.42x10 ⁻⁷

The graded zirconia and the zirconia, titania, yttria blend recorded coefficients of friction values between 0.15-0.38 and average wear coefficients of 10⁻⁶ and 10⁻⁷ mm³/N-m running against the ceramic counterface.

In summary, the friction and wear screening of the plasma sprayed coatings identified the chromia-silica composite, high carbon iron-molybdenum alloy and the self-lubricating PS212 as candidates for further friction and wear optimization. These coatings exhibited coefficients of friction and average wear coefficients which met the goals of this program. The cobalt-chromium alloy with TRIBALLOY intermetallic additions, the high molybde-

num-nickel-chromium blend, and the nickel-chromium-molybdenum blend exhibited either coefficient of friction values above 0.2 or average wear coefficients above 10^{-6} mm³/N-m running against either the ceramic or metallic counterface. For these reasons, these coatings were not chosen for further optimization.

3.3.3 Candidate Wear Resistant Chemical/Physical Vapor Deposited Coatings

Two chemical vapor deposited coatings, chrome carbide and a layered titanium carbide/titanium nitride, were applied to H-13 tool steel substrates and evaluated. A mid-temperature chemical vapor deposited titanium carbonitride coating was also evaluated. This coating, however, was applied to a 17-4 stainless steel substrate. Finally, a low temperature arc vapor deposited (LTAVD) chrome nitride coating was applied to a cast iron substrate and evaluated. Tables 3.3.1, 3.3.2, and 3.3.12-15 contain the results of the pin-on-disk friction and wear screening for these coatings.

The layered titanium carbide/titanium nitride coating recorded low coefficient of friction values (0.08-0.18), and average disk and counterface wear coefficients (10^{-8} and 10^{-9} mm³/N-m) running against the ceramic counterfaces. Slightly higher coefficient of friction values (0.18-0.22) were obtained running this layered coating against the metallic counterface. These higher coefficient of friction values resulted in higher average wear coefficients (10^{-6} mm³/N-m).

High coefficient of friction values (0.09-0.59) and average wear coefficients (10^{-7} mm³/N-m) were obtained running the chemical vapor deposited chrome carbide coating against the ceramic counterfaces. Running this coating against a metallic counterface also produced very high coefficients of friction (0.24-0.31) as well as unacceptable average disk and counterface wear coefficients (10^{-5} and 10^{-6} mm³/N-m).

Figures 3.3.3 and 3.3.4 show that the only wear apparent on either CVD coating, after testing, was a small amount of polishing of the coating surfaces.

Very low average wear coefficients (10^{-8} to 10^{-9} mm³/N-m) were obtained running the mid-temperature chemical vapor deposited titanium carbonitride coating against either ceramic counterface. The coefficient of friction values for these specimens ranged from 0.09 to 0.15. Figure 3.3.5 reveals that polishing by the ceramic counterface was the only wear observed after the test. High coefficient of friction values (0.24) and very high disk and counterface average wear coefficients (10^{-6} mm³/M-n) were obtained running the titanium carbonitride coating against a metallic counterface.

FIGURE 3.3.3
SEM PHOTOMICROGRAPHS OF LAYERED
TITANIUM NITRIDE/TITANIUM CARBIDE
AFTER PIN-ON-DISK TESTING

MAGNIFICATION 50X

MAGNIFICATION 1000X

FIGURE 3.3.4
SEM PHOTOMICROGRAPHS OF
CVD CHROME CARBIDE AFTER PIN-ON-DISK TESTING

MAGNIFICATION 50X

1000X MAGNIFICATION OF REGION 21

FIGURE 3.3.5
SEM PHOTOMICROGRAPHS OF
MID-TEMPERATURE CVD TITANIUM CARBO-NITRIDE
AFTER PIN-ON-DISK TESTING

MAGNIFICATION 50X

1000X MAGNIFICATION OF REGION 30

Excellent coefficient of friction values (0.08-0.19) and average wear coefficients (10^{-8} mm³/N-m) were obtained running the low temperature arc vapor deposited chrome nitride against the ceramic counterface. Coefficient of friction values ranging from 0.01-0.11 and average wear coefficients of 10^{-7} mm³/N-m were recorded running the chrome nitride against the metallic counterface.

The chemical vapor deposited tungsten/tungsten carbide specimens had a 10 to 50 microinch surface roughness. This rough surface finish caused high coefficients of friction values (0.15-0.21) when this coating was run against either counterface. These high coefficient of friction values, in turn, caused wear coefficients, ranging from 10^{-6} to 10^{-7} mm³/N-m. However, most of the wear occurred at the start of testing; as the tungsten/tungsten carbide surface "wore" and became smoother, the coefficient of friction decreased to below 0.2.

The mid-temperature chemical vapor deposited titanium carbonitride, the low temperature arc vapor deposited chrome nitride and the layered titanium carbide/ titanium nitride meet the coefficient of friction and average wear coefficient goals of this program. For this reason, these coatings all are candidates for future friction and wear optimization. The chemical vapor deposited chrome carbide and tungsten/tungsten carbide coatings were not selected for further friction and wear optimization since these coatings recorded coefficient of friction values greater than 0.2 and wear coefficients above 10^{-7} mm³/N-m.

3.3.4 Wear Resistant Enamel Coatings

Two wear resistant enamel coating compositions and approaches were evaluated. One approach, taken by Solar Turbine, was the addition of hard particles to their proprietary Vitreous Phase Coating composition (VPC). The second approach, used by the University of Illinois, involved plasma spraying the hard particles on top of an A.I. Andrew's cobalt based enamel composition.

Solar was able to load their VPC with up to 80 volume percent oxide based hard particle additions without significantly decreasing the adherence of the VPC to the metallic substrate.

The effect of 50 and 70 volume percent hard particle additions on the friction and wear properties of the VPC were determined first. Solar prepared the following VPC compositions: the VPC without any hard particle additions (base composition), the VPC with a 50 volume percent aluminum oxide hard particle addition, the VPC with 50 and 70 volume percent chrome oxide hard particle additions, and the VPC with a 50 volume percent zirconium oxide hard particle addition. The results of this initial screening are contained in Table 3.3.5.

The base Solar VPC composition without any hard particle additions exhibited high friction values (0.11-0.44) and wear coefficients (10^{-5} mm³/N-m) running against either the ceramic or metallic counterface. For this reason, these tests ran only for 30 and 60 minutes respectively. Representative photomicrographs of these wear surfaces, after testing, are contained in Figure 3.3.6. These photomicrographs show that, the VPC pulverized and spalled under the compressive load applied by the ceramic counterface. The region labeled N in Figure 3.3.7 shows a wear track location on the disk, where coating spallation had occurred during the test. Also observed within this wear track was a thick, black residue (region labeled D).

The 50 or 70 volume percent chrome oxide, 50 volume percent aluminum oxide or 50 volume percent zirconium oxide hard particle additions to the base VPC composition did not improve the wear properties of the coating. Coefficient of friction values for these specimens ranged from 0.13, running against the metallic counterface to above 0.5, running against the ceramic counterface. The average wear coefficients, running against the ceramic counterface were in the 10^{-5} mm³/N-m range. Slightly improved wear coefficients, 10^{-7} mm³/N-m, were obtained running these coatings against the metallic counterface.

Table 3.3.5
Pin-on-Disk Friction & Wear Test Results--Hard Particle
Additions to Vitreous Phase Coating (VPC)

VPC Composition	Pin	Test Time (min.)	Friction		Average Wear Coefficient (mm ³ /N-m)	
			Min.	Max.	Coating	Pin
Base comp.	ZrO ₂	30	0.20	0.37	7.31×10^{-5}	2.24×10^{-7}
	ZrO ₂	30	0.26	0.44	4.57×10^{-5}	1.27×10^{-7}
	440 SS	60	0.11	0.15	3.50×10^{-7}	2.05×10^{-6}
Base + 50 vol.% Al ₂ O ₃	ZrO ₂	30	0.18	0.18	6.48×10^{-7}	4.71×10^{-6}
	440 SS	1	0.55	0.55	4.47×10^{-5}	3.25×10^{-6}
Base + 50 vol.% Cr ₂ O ₃	ZrO ₂	3	0.42	0.53	3.57×10^{-5}	1.32×10^{-8}
	ZrO ₂	3	0.13	0.53	5.60×10^{-4}	7.45×10^{-6}
Base - 70 vol.% Cr ₂ O ₃	ZrO ₂	48	0.13	0.59	6.33×10^{-7}	1.98×10^{-7}
Base + 50 vol.% ZrO ₂	ZrO ₂	30	0.22	0.38	8.33×10^{-5}	1.42×10^{-7}
	ZrO ₂	60	0.14	0.18	5.61×10^{-7}	2.15×10^{-6}

Figure 3.3.7. displays the wear tracks of the VPC plus 50 and the VPC plus 70 volume percent hard particle additions after testing. These photomicrographs show that portions of the VPC plus hard particle coatings were completely worn to the metallic substrate during the testing. The addition of the hard particles did not improve the average wear coefficient of the VPC coating.

FIGURE 3.3.6
SURFACE OF SOLAR VPC ENAMEL AFTER TESTING

A. MAGNIFICATION 1.5X

B. 10X MAGNIFICATION OF REGION B

A black residue also was observed within the wear track of these specimens. This undesirable chemical reaction was the reason it was decided to discontinue the development work with the VPC enamel and switch to the A.I. Andrew's cobalt based enamel composition.

The process used by the University of Illinois to produce wear resistant enamel coatings was to plasma spray hard particles on top of an A.I. Andrew's cobalt based enamel. In general, the processing for these specimens consisted of slurry spraying an enamel coating on the specimen, firing to fuse the enamel, and then plasma spraying hard particles on top of the enamel to improve the wear properties of the enamel. The University of Illinois was able to successively plasma spray chrome carbide, chrome oxide and zirconia hard particles on top of the enamel. These coatings had their friction and wear properties evaluated running against both ceramic and metallic counterfaces a pin-on-disk test. Table 3.3.18 contain the results of this testing.

Very high friction coefficients (0.25) were encountered running the plasma sprayed zirconia against the zirconia counterface. Large portions of the plasma sprayed zirconia coating spalled after only five minutes of testing and the test was terminated.

The chrome carbide plasma sprayed specimens recorded coefficient of friction values between 0.09-0.24 running against all three ceramic counterfaces. Although these coatings had relatively low coefficient of friction values, the average wear coefficients were all greater than 10^{-7} mm³/N-m. For this reason, these specimens ran only 48, 60 and 120 minutes against either ceramic counterface before a 2.5 mm wear scar developed. Running against the metallic counterface, the plasma sprayed chrome carbide recorded higher coefficient of friction values (0.24 and 0.31) and average wear coefficients (10^{-6} mm³/N-m). This specimen ran only 30 minutes before a 2.5 mm wear scar developed.

Running the zirconia counterface against the chrome oxide plasma sprayed over the enamel resulted in coefficient of friction values between 0.09-0.18, and average wear coefficients of between 10^{-6} to 10^{-7} mm³/N-m. One specimen ran only 30 minutes, while the remaining two specimens ran for 120 minutes before developing a 2.5 mm wear scar.

This pin-on-disk testing showed that plasma spraying hard particles on top of an enamel bond coating would not produce coating with acceptable coefficient of friction and/or wear coefficients.

The University of Illinois produced another specimen by sprinkling silicon carbide particles on top a molten A.I. Andrews' enamel. This specimen had an extremely rough surface texture and, for this reason, had coefficient of friction values of 0.30 and average wear coefficients for both the coating and counterface of 10^{-4} mm³/N-m.

FIGURE 3.3.7
SURFACE OF SOLAR VPC ENAMEL TESTING
CONTAINING CHROME OXIDE PARTICLES AFTER TESTING
(MAGNIFICATION 10X)

A. 50 VOLUME PERCENT CHROME OXIDE ADDITION

B. 70 VOLUME PERCENT CHROME OXIDE ADDITION

It was decided to evaluate lower volume loadings of the hard particle additions to the enamel. Hopefully, with the lower volume loading of hard particles there would be an increased amount of enamel coating around each particle improving the bonding of the particles into the enamel.

Enamel compositions containing 10 and 20 weight percent zirconium oxide, and 10 and 20 percent chrome oxide hard particle additions were processed by the University of Illinois. The friction and wear properties of these specimens were then evaluated by running against a zirconia counterface in a lubricated pin-on-disk test. Irrespective of the hard particle composition or volume loading, the zirconia counterface wore a 2.5 mm wear scar in the enamel coating after only five minutes of testing.

Table 3.3.6
Pin-on-Disk Friction & Wear Test Results--Hard Particle
Additions to Cast Iron Porcelain Enamel

Coating	Pin	Test Time (min.)	Friction		Average Wear Coefficient (mm ³ /N-m)	
			Min.	Max.	Coating	Pin
Plasma Sprayed	ZrO ₂	120	0.09	0.18	1.13x10 ⁻⁷	2.25x10 ⁻⁶
Chrome Carbide	ZrO ₂	120	0.09	0.15	5.34x10 ⁻⁷	2.44x10 ⁻⁷
	ZrO ₂	120	0.07	0.20	2.03x10 ⁻⁷	2.39x10 ⁻⁷
	440 SS	30	0.15	0.15	2.02x10 ⁻⁷	7.38x10 ⁻⁶
	440 SS	120	0.09	0.31	8.61x10 ⁻⁸	1.39x10 ⁻⁷
Plasma Sprayed	ZrO ₂	30	0.09	0.14	1.06x10 ⁻⁶	4.12x10 ⁻⁶
Chrome Oxide	ZrO ₂	120	0.07	0.16	6.44x10 ⁻⁷	8.82x10 ⁻⁸
Plasma Sprayed ZrO ₂ **	ZrO ₂	1	0.25	---	---	---
Sic Sprinkled on Molten Enamel	ZrO ₂	30	0.11	0.37	2.01x10 ⁻⁴	2.53x10 ⁻⁵

** coating developed a 2.5 mm wear scar in less than 2 minutes of testing

Finally, the University of Illinois attempted to plasma spray an A.I. Andrews' enamel composition containing a 10 volume percent zirconium oxide hard particle addition. This plasma coating contained approximately 50 volume percent porosity and had a very rough surface finish. The combination of 50 high porosity and very rough surface finish caused this specimen to develop a 2.5 mm wear scar after only one minute of testing when run against a zirconia counterface.

3.3.5 Candidate Piston Ring and Cylinder Liner Coating Pairs Selected for Further Optimization

Three plasma spray coatings, one mid-temperature chemical vapor deposited and one LTAVD coating were selected for further optimization. Two of the plasma spray coatings, the chromia-silica and the high carbon iron-molybdenum, were chosen because both recorded low friction and wear coefficients during the initial pin-on-disk screening running against either the zirconia or 440C stainless steel counterfaces. Also selected for further optimization, was the plasma sprayed PS212. The PS212 was selected even though its wear performance was only marginal; NASA had previously published some promising results using the PS212 coating on cylinder liners of a Sterling engine running unlubricated at elevated temperatures.

The mid-temperature titanium carbonitride chemical vapor deposited coating was selected as a potential wear resistant piston ring coating because it exhibited low friction and wear coefficients running against the zirconia counterface. In addition, it should not require machining after application to obtain the desired surface finish, offering a potential cost advantage over a plasma sprayed coating.

Excellent friction and wear results were obtained with the LTAVD chrome nitride coating running against either the zirconia or 440C stainless steel counterfaces. These promising results make this coating a candidate wear coating for cylinder liners. In addition, the unique LTAVD application process enables the chrome nitride coating to be uniformly applied to the bores of components.

3.4 Optimization of Coatings for Wear and Friction

3.4.1 Description of Hohman A-6 Friction and Wear Test Procedure

The Hohman A-6, Double Rub Shoe Machine was selected for the optimization of the most promising wear resistant coatings identified from the pin-on-disk screening. The Hohman A-6 has been used in laboratories for 35 years for evaluation of coatings. In general, the Hohman A-6 test (Figure 3.4.1) loads two coated shoe specimens against the circumference of a coated rotating disk specimen. Area contact is obtained by designing the shoes to conform to the surface of the disk. In this program, the candidate piston ring wear coatings were applied to the inner diameter of both shoes while the candidate cylinder liner coatings were applied to the outer diameter of the disks.

All the Hohman testing was performed at 350 C under lubricated conditions. This test temperature was obtained by surrounding the specimens with electric resistant heating elements embedded within insulating blocks to maintain the desired 350 C temperature.

An 89 Newton normal load was applied with a surface velocity of 3.4 m/sec. An experimental synthetic lubricant from Lubrizol was used for all the friction and wear optimization. The lubricant was delivered to the coated shoes by a peristaltic pump at a rate of 3 ml/min. through two hypodermic syringes, placed on either side of the shaft, to allow the lubricant to drip uniformly onto the contact surfaces.

Figure 3.4.1

The criteria used for selecting the candidate piston ring or cylinder liner coating pairs was:

1. The ability of the process used for applying the coating to coat the desired surface configuration (i.e., I.D. of a cylinder liner or the O.D. of a piston ring),
2. The coating can be applied to the metallic substrate using a process which will not degrade the mechanical properties of the substrate,
3. The coating will have tensile pull adherence values above 9.7 MPa when applied to cast iron, for cylinder liner candidates, and H-13 tool steel or 17-4 PH stainless steel for piston ring candidates and,
4. The pin-on-disk friction and wear results run against both the metallic and ceramic counterfaces.

The following wear resistant piston ring coatings were selected to be evaluated against candidate cylinder liner coatings: the plasma sprayed high carbon iron-molybdenum blend,

the plasma sprayed chromia-silica composite, the NASA self-lubricating PS212 and the mid-temperature chemical vapor deposited titanium carbo-nitride. Wear resistant cylinder liner coatings selected included the plasma sprayed high carbon iron-molybdenum blend, the plasma sprayed chromia-silica composite and the low temperature arc vapor deposited chrome nitride.

3.4.2 Friction and Wear Test Results

After selection of the candidate piston ring and cylinder liner coatings, a Hohman A-6 test matrix was developed for optimization of the friction and wear properties of each piston ring/cylinder liner pair. The test matrix consisted of running the four candidate piston ring coatings against each of the three candidate cylinder liner coatings. This test matrix consisted of 12 coating pairs. Tables 3.4.1, 3.4.2, and 3.4.3 contain the results of the Hohman A-6 friction and wear optimization.

Running plasma sprayed high carbon iron-molybdenum coated rub shoes against a plasma sprayed chromia-silica coated disk (Table 3.4.1) produced coefficient of friction values below 0.15 and wear coefficients ranging from 2.3 to $0.4 \times 10^{-8} \text{ mm}^3/\text{N-m}$ for the shoes and from 2.43 to $14.2 \times 10^{-8} \text{ mm}^3/\text{N-m}$ for the disks. The Hohman test results obtained from this this coating pair showed that this piston ring/cylinder liner coating pair would meet the program goals for both friction and wear.

Scanning electron photomicrographs of the surfaces, after 900 hours of Hohman A-6 friction and wear testing, of one set of the high carbon iron-molybdenum rub shoes and the corresponding chromia-silica coated disk are contained in Figures 3.4.2.A and 3.4.2.B. These photomicrographs show that during the test, the sliding motion of the rub shoes over the disk developed tensile stresses within these coatings and these tensile stresses initiated shallow subsurface cracking. The average wear coefficient was less than $10^{-8} \text{ mm}^3/\text{N-m}$.

Coefficient of friction values of above 0.2 were obtained by running plasma sprayed PS 212 rub shoes against a plasma sprayed chromia-silica disk. These high coefficient of friction values, in turn, caused high PS 212 wear coefficients and, for this reason, this coating pair ran only 480 hours before the chromia-silica coating completely wore through the PS 212. Table 3.4.1 lists average wear coefficients of 9.11×10^{-9} for the chromia-silica and $1.70 \times 10^{-7} \text{ mm}^3/\text{N-m}$ for the PS212 coatings. Scanning electron photomicrographs of this coating pair after testing, Figures 3.4.3.A and 3.4.3.B, showed extensive galling on both PS212 coated rub shoes but very little wear on the corresponding chromia-silica disk. Thus, during the test, the chromia-silica coating abraded the solid lubricants located within the chrome carbide matrix. Although the solid lubricants were abraded during the test, there was no significant decrease in friction coefficient.

Table 3.4.1
Hohman A-6 Friction and Wear Results
Candidate Piston Ring Coatings Run Against
Chromia-Silica Coated Cylinder Liner Coating

Candidate Piston Ring Coating	Time (Min.)	Average Wear Coefficient (mm ³ /N-m) Piston Ring	Liner
Chromia-Silica	120	1.70x10 ⁻⁹	3.36x10 ⁻⁶
PS212	480	7.61x10 ⁻⁷	9.11x10 ⁻⁹
Mid-temperature Ti(C,N)	55	3.50x10 ⁻⁹	1.52x10 ⁻⁵
High Carbon Iron-Molybdenum	370	2.30x10 ⁻⁸	2.43x10 ⁻⁸
	975	7.56x10 ⁻⁸	1.66x10 ⁻⁸
	960	4.14x10 ⁻⁸	5.91x10 ⁻⁸
	899	4.48x10 ⁻⁹	1.42x10 ⁻⁷

SUMMARY

-The High Carbon Iron-Molybdenum piston ring coating running against the chromia-silica cylinder liner pair was the only coating pair which met the program wear coefficient goals.

-Unacceptable Chromia-Silicate wear was observed when this cylinder liner coating was run against either the Chromia-Silica or Mid-temperature Ti(C,N) piston ring coatings.

-Unacceptable PS212 piston ring coating wear occurred when this coating was run against the Chromia-Silica coating.

Table 3.4.2
Hohman A-6 Friction and Wear Results
Candidate Piston Ring Coatings Run Against a
LTAVD Chrome Nitride Cylinder Liner Coating

Candidate Piston Ring Coating	Time (Min.)	Average Wear Coefficient (mm ³ /N-m) Piston Ring	Liner
Chromia-Silica	8	2.17x10 ⁻⁸	8.36x10 ⁻⁷
	5	2.17x10 ⁻⁷	6.95x10 ⁻⁶
PS212	120	7.86x10 ⁻⁹	1.04x10 ⁻⁴
Mid-temperature Ti(C,N)	6	1.72x10 ⁻⁷	1.05x10 ⁻⁶
	5	2.95x10 ⁻⁷	6.35x10 ⁻⁶
	30	4.03x10 ⁻⁹	7.72x10 ⁻⁶
High Carbon Iron-Molybdenum	120	1.23x10 ⁻⁹	3.13x10 ⁻⁷
	650	2.85x10 ⁻⁸	1.92x10 ⁻⁸
	375	7.16x10 ⁻⁸	1.48x10 ⁻⁸

SUMMARY

-The High Carbon Iron-Molybdenum running against the Chrome Nitride was the only coating pair which met the programs wear coefficient goals.

-Unacceptable Chrome Nitride wear was observed running against the following candidate piston ring coatings: PS212, Chromia-Silica, and Mid-temperature Ti(C,N).

FIGURE 3.4.2A
SURFACES AFTER HOHMAN A-6 TESTING
TEST 44--PLASMA SPRAYED HIGH CARBON IRON-MOLYBDENUM
COATED SHOES RUNNING AGAINST A
PLASMA SPRAYED CHROMIA-SILICA DISK

CHROMIA-SILICA DISK

MAGNIFICATION 100X

REGION 045 MAGNIFICATION 500X

FIGURE 3.4.2B
SURFACES AFTER HOHMAN A-6 TESTING
TEST 44--PLASMA SPRAYED HIGH CARBON IRON-MOLYBDENUM
COATED SHOES RUNNING AGAINST A
PLASMA SPRAYED CHROMIA-SILICA DISK

HIGH CARBON IRON-MOLYBDENUM SHOES

MAGNIFICATION 100X

REGION 110 MAGNIFICATION 500X

FIGURE 3.4.3A
SURFACES AFTER HOHMAN A-6 TESTING
TEST 45--PLASMA SPRAYED PS212 SHOES RUNNING
AGAINST A PLASMA SPRAYED CHROMIA-SILICA COATED DISK

CHROMIA-SILICA DISK

MAGNIFICATION 100X

MAGNIFICATION 500X

FIGURE 3.4.3B
SURFACES AFTER HOHMAN A-6 TESTING
TEST 45--PLASMA SPRAYED PS212 SHOES RUNNING
AGAINST A PLASMA SPRAYED CHROMIA-SILICA COATED DISK

PS212 COATED SHOES

MAGNIFICATION 100X

MAGNIFICATION 500X

Table 3.4.3
Hohman A-6 Friction and Wear Results
Candidate Piston Ring Coatings Run Against A High Carbon
Iron-Molybdenum Liner Coating

Candidate Piston Ring Coating	Time (Min.)	Average Wear Coefficient (mm ³ /N-m)	
		Piston Ring	Liner
Mid-temperature Ti(C,N)	34	9.55x10 ⁻⁸	1.68x10 ⁻⁸
	241	1.42x10 ⁻⁹	1.56x10 ⁻⁸
LTAVD Chrome Nitride (Coating Thickness 5 microns)	168	2.79x10 ⁻⁹	1.06x10 ⁻⁹ (1)
	120	----	5.60x10 ⁻⁹ (2)
Chromia-Silica	120	< 10 ⁻⁹	7.72x10 ⁻¹⁰

SUMMARY

-The Mid-temperature Ti(C,N) coating worn through the High Carbon Iron-Molybdenum coating were very rapidly and for this reason this coating pair was dropped from the program.

-Again the Chromia-Silica running against the High Carbon Iron-Molybdenum was an acceptable coating pair.

Notes:

1. CrN coating spalled during heat-up after initial wear measurements were obtained.
2. Spallation of CrN coating occurred before the initial wear measurements were taken.

Running plasma sprayed chromia-silica or mid-temperature chemical vapor deposited titanium carbonitride coated rub shoes against the plasma sprayed chromia-silica coated disks produced friction coefficients above 0.2 and average disk wear coefficients ranging from 10⁻⁵ to 10⁻⁶ mm³/N-m, respectively. Both the friction and disk wear coefficients for both these coating pairs were above the program goals.

Scanning electron photomicrographs of these coating pairs, after testing, are contained in Figures 3.4.4 and 3.4.5. These photomicrographs showed extensive cracking and large regions of coating delamination on both the chromia-silica disks. A lesser amount of cracking and coating delamination was observed on the corresponding chromia-silica or titanium carbonitride coated rub shoes.

Another coating pair which met the program's friction and wear goals was plasma sprayed high carbon iron-molybdenum rub shoes running against a low temperature arc vapor deposited (LTAVD) chrome nitride coated disk (Table 3.4.2). This coating pair had coefficient of friction values below 0.15 and, in three tests, had average wear coefficients which ranged from 1.23x10⁻⁹ to 7.16x10⁻⁸ mm³/N-m for the high carbon iron-molybdenum coated shoes and from 3.13x10⁻⁷ to 1.48x10⁻⁸ mm³/N-m for the chrome nitride coated disk.

The scanning electron photomicrographs of this coating pair after 650 hours of testing, are contained in Figures 3.4.6.A and 3.4.6.B. Extensive cracking and smearing were characteristic of the high carbon iron-molybdenum coating after testing. Also observed within the coating were small isolated regions where some coating delamination had occurred. Very little chrome nitride coating was present on the disk after testing.

FIGURE 3.4.4A
SURFACES AFTER HOHMAN A-6 TESTING
TEST 42--PLASMA SPRAYED CHROMIA-SILICA COATED SHOES
RUNNING AGAINST A PLASMA SPRAYED CHROMIA-SILICA DISK

CHROMIA-SILICA COATED DISK

MAGNIFICATION 100X

MAGNIFICATION 500X

FIGURE 3.4.4B
SURFACES AFTER HOHMAN A-6 TESTING
TEST 42--PLASMA SPRAYED CHROMIA-SILICA COATED SHOES
RUNNING AGAINST A PLASMA SPRAYED CHROMIA-SILICA DISK

CHROMIA-SILICA COATED DISK

MAGNIFICATION 50X

REGION 052

MAGNIFICATION 500X

FIGURE 4.3.5A
SURFACES AFTER HOHMAN A-6 TESTING
TEST 49--MID-TEMPERATURE Ti (C,N) CVD COATED SHOES RUNNING
AGAINST A PLASMA SPRAYED CHROMIA-SILICA COATED DISK

CHROMIA-SILICA DISK

MAGNIFICATION 100X

REGION 013 MAGNIFICATION 500X

FIGURE 4.3.5B
SURFACES AFTER HOHMAN A-6 TESTING
TEST 49--MID-TEMPERATURE Ti (C,N) CVD COATED SHOES RUNNING
AGAINST A PLASMA SPRAYED CHROMIA-SILICA COATED DISK

CHROMIA-SILICA DISK

MAGNIFICATION 100X

REGION 57 MAGNIFICATION 500X

FIGURE 3.4.6A
SURFACES AFTER HOHMAN A-6 TESTING
TEST 45--PLASMA SPRAYED HIGH CARBON IRON-MOLYBDENUM
SHOES RUNNING AGAINST A LTAVD CHROME NITRIDE COATED DISK

CHROME NITRIDE COATED DISK

MAGNIFICATION 100X

REGION 001 MAGNIFICATION 500X

FIGURE 3.4.6B
SURFACES AFTER HOHMAN A-6 TESTING
TEST 43--PLASMA SPRAYED HIGH CARBON IRON-MOLYBDENUM
SHOES RUNNING AGAINST A LTAVD CHROME NITRIDE COATED DISK

HIGH CARBON IRON-MOLYBDENUM COATED SHOES

MAGNIFICATION 100X

REGION 067 MAGNIFICATION 500X

Running either plasma sprayed chromia-silica or mid-temperature titanium carbonitride CVD rub shoes against the LTAVD chrome nitride coated disk resulted in coefficient of friction values above 0.20 and very high chrome nitride average wear coefficients (10^{-6} mm³/N-m). For this reason, these tests were terminated after only 5-30 minutes of testing because the shoes wore completely through the chrome nitride coating. Scanning electron photomicrographs of one set of these specimens (Figures 3.4.7.A and 3.4.7.B), after testing, showed very little wear on the mid-temperature titanium carbonitride rub shoes, while the chrome nitride coating was completely worn from the disk.

Average disk wear coefficients of 1.04×10^{-4} mm³/N-m were obtained running plasma sprayed PS 212 coated rub shoes against a chrome nitride coated disk. This wear coefficient was well above the desired 10^{-8} mm³/N-m wear coefficient of this program. High friction coefficients were the probable reason for the poor average wear coefficient of the PS 212.

The Hohman friction and wear results for the mid-temperature titanium carbonitride, LTAVD chrome nitride and chromia-silica coated rub shoes running against plasma sprayed high carbon iron-molybdenum coated disks are contained in Table 3.4.3. Running the mid-temperature titanium carbonitride rub shoes against the high carbon iron-molybdenum disk resulted in friction coefficients of below 0.15, and average wear coefficients of 1.42×10^{-9} to 9.55×10^{-8} mm³/N-m for the titanium carbonitride coated shoes and 1.56×10^{-8} to 1.86×10^{-8} mm³/N-m for the high carbon iron-molybdenum shoes. These results indicate that this coating pair will meet the friction and wear goals of this program.

Wear testing of the 5 micron thick chrome nitride coated rub shoes against the high carbon iron-molybdenum coated disks could not be completed because of chrome nitride coating spallation. The first instance of spallation occurred during a heat-up cycle after the test was stopped to obtain an initial set of wear measurements. Prior to the catastrophic coating failure both the rub shoe and disk had average wear coefficients of 10^{-9} mm³/N-m. The second instance of coating spallation occurred prior to testing when the rub shoes were heated to the 350 C test temperature.

Friction coefficients below 0.15 and average wear coefficients of less than 10^{-9} mm³/N-m were obtained for both the rub shoes and the disk running plasma sprayed chromia-silica rub shoes against a high carbon iron-molybdenum disk. The friction and wear coefficients obtained from this coating pair were similar to those obtained running high carbon iron-molybdenum shoes against a chromia-silica disk (Table 3.4.1).

FIGURE 3.4.7A
SURFACES AFTER HOHMAN A-6 TESTING
TEST 50--MID-TEMPERATURE Ti (C,N) COATED SHOES RUNNING
AGAINST A LTAVD CHROME NITRIDE COATED DISK

CHROME NITRIDE COATED DISK

MAGNIFICATION 100X

MAGNIFICATION 500X

FIGURE 3.4.7B
SURFACES AFTER HOHMAN A-6 TESTING
TEST 50--MID-TEMPERATURE Ti (C,N) COATED SHOES RUNNING
AGAINST A LTAVD CHROME NITRIDE COATED DISK

MID-TEMPERATURE Ti (C,N) SHOES

MAGNIFICATION 200X

MAGNIFICATION 500X

3.4.3 Selection of Piston Ring/ Cylinder Liner Wear Resistant Coating Pairs Which Meet the Program Goals

The friction and wear optimization identified two wear resistant piston ring/cylinder liner coating combinations which met both the friction and wear goals of this program. The two coating pairs were:

1. Plasma sprayed high carbon iron-molybdenum coated piston rings and plasma sprayed chromia-silica cylinder liner, and
2. Plasma sprayed high carbon iron-molybdenum coated piston rings and low temperature arc vapor deposited chrome nitride.

Both these coating combinations exhibited coefficient of friction values 0.2 or below and had average wear coefficients of less than 10^{-8} mm³/N-m.

3.5 Characterization of Candidate Wear Resistant Piston Ring/Cylinder Liner Coating Pairs Which Demonstrated Excellent Friction and Wear Performance

The two wear resistant piston ring and cylinder liner coating pairs identified as meeting the friction and wear goals of this program were next fully characterized for wear, thermal shock resistance and environmental stability in a diesel exhaust atmosphere.

3.5.1 Development of Wear Curves

Wear curves were obtained for the high carbon iron-molybdenum running against the chromia-silica and the high carbon iron-molybdenum running against the chrome nitride.

These wear curves were obtained by running the specimens on the Hohman A-6 Double Rub-Shoe machine, at 350 C, with a normal load of 89 N, and a surface velocity of 3.4 m/sec. During the testing, an experimental synthetic lubricant from Lubrizol was used. The lubricant was delivered to each shoe surface using a peristaltic pump at a rate of 3ml/min. After every 120 minutes the test was interrupted to measure the wear on both the shoe and disk surfaces. Wear curves for each coating pair were then developed from these wear measurements. The wear curves for the high carbon iron-molybdenum running against the chromia-silica are contained in Figures 3.5.1 and 3.5.2. Figures 3.5.3 and 3.5.4 contain the wear curves for the high carbon iron-molybdenum running against the chrome nitride.

The wear scar volumes measured on the high carbon iron-molybdenum coated rub shoes after 980 hours of friction and wear testing against the chromia-silica coated disks were 0.55, 0.27 and 0.05 mm³, for Tests 56, 70, and 72, respectively (Figure 3.5.1). The wear

scars measured on two of the corresponding chromia-silica coated disks from Tests 56 and 70 were 0.05 and 2.0 mm³. The third chromia-silica coated disk (Test 72) had a wear scar volume greater than 8.0 mm³. However, over 50 percent of the volume of this wear scar occurred after 380 minutes of testing when one of the rub shoes "slipped" off the disk. Eliminating the wear which occurred when the rub shoe "slipped," the volume of the wear scar would have been less than 4.0 mm³. The volumes of the wear scars measured on both the shoes and disks after 980 hours of testing indicated that this coating pair, a high carbon iron-molybdenum coated piston ring and a chromia-silica coated cylinder liner, will have wear rates which would meet the commercial durability requirements.

The wear curves obtained from running the high carbon iron-molybdenum coated rub shoes against a low temperature arc vapor deposited chrome nitride coated disk showed that very little wear occurred on either the rub shoes or the disk. One set of high carbon iron-molybdenum shoes exhibited essentially no wear after 680 hours of testing (Test 43) while the second set had a 0.2 mm³ wear scar after 380 hours of testing, Test 54. However, the majority of the wear occurred during the initial 100 hours of testing and after this break-in period very little additional wear was seen on these shoes. The wear scars measured on the corresponding chrome nitride coated disks were 0.1 mm³ or less. Although the wear scars on the chrome nitride disks were very small, the tests had to be terminated after 650 hours and 380 hours because the high carbon iron-molybdenum coated shoes had worn completely through the 3 micron thick chrome nitride coating. These wear curves show that a three micron thick chrome nitride coating, although it meets the program goals with respect to both friction and wear, will not meet the commercial durability goals. A 15 to 20 micron thick chrome nitride coating is required to meet these commercial durability goals.

Once the wear curves showed that a thicker chrome nitride coating is required a second set of chrome nitride specimens was obtained. These specimens were coated using the same process conditions as the initial specimens except the chrome nitride was applied to a thickness of 5 microns. Wear curves for these specimens could not be obtained because the chrome nitride coating spalled completely from the disk. On one specimen the spallation occurred during the initial heat to 350 C prior to initiating the wear test. The second specimen spalled after 120 minutes of wear testing during the heat-up to 350 C after the initial set of wear measurements was obtained.

3.5.2 Oxidation Resistance

The oxidation resistance of the plasma sprayed high carbon iron-Molybdenum and plasma sprayed chromia-silica coatings was evaluated by exposing coated cast iron Falex disks to

a simulated diesel exhaust atmosphere, for 500 hours at 400 C, in a tube furnace. The simulated diesel atmosphere was obtained from the mixing of various gases. The composition of this simulated diesel exhaust atmosphere was:

Oxygen (O_2) 9.6%
Carbon Dioxide (CO_2) 8.3%
Carbon Monoxide (CO) 1439 ppm
Nitric Oxide (NO) 1439 ppm
Hydrocarbons 264 ppm
Water Vapor (H_2O) 7.3%
Nitrogen (N_2) Balance.

Figure 3.5.1
Wear Curve for High Carbon Iron Molybdenum Shoes
Running Against a Chromia-Silica Disk

Figure 3.5.2
Wear Curve for Chromia-Silica Disk Run Against High Carbon Iron-Molybdenum Shoes

Figure 3.5.3 Wear Curve for High Carbon Iron-Molybdenum Shoes Running Against a Chrome Nitride Disk

Figure 3.5.4 Wear Curve for Chrome Nitride Disk Running Against High Carbon Iron Shoes

The plasma sprayed chromia-silica coating was not affected by the simulated diesel exhaust atmosphere. No coating spallation or discoloration was observed on any of the five specimens exposed. In fact, the physical appearance of these specimens was identical to the physical appearance of plasma sprayed chromia-silica specimens which had not been exposed to the simulated diesel exhaust atmosphere.

The high carbon iron-molybdenum plasma sprayed coated discolored during the 500 hour exposure to the simulated diesel exhaust atmosphere. This discoloration was caused by oxidation of the iron on the surface of the specimens which was in direct contact with the atmosphere. The oxidation was limited to the very top surface of the coating and for this reason no significant increase in coating thickness or spallation occurred.

3.5.3 Thermal Shock Resistance

The thermal shock resistance of the plasma sprayed high carbon iron-molybdenum and plasma sprayed chromia-silica coatings was determined by quenching plasma sprayed cast iron Falex specimens fifty (50) times from 650 C into boiling water. Each specimen was thermocoupled to assure that it attained a 650 C temperature prior to each quench. The thermocouple also illustrated that it took approximately seven seconds for the Falex disks to be quenched from 650 to 100 C. Representative photomicrographs of two specimens after completion of the thermal shock testing are contained in Figures 3.5.5 and 3.5.6.

The photomicrographs show that cracks developed in both plasma coatings. These cracks were first noticed after three thermal shock cycles. Although some additional crack propagation occurred during the subsequent thermal shock cycles, no coating disbonding or coating spallation occurred. These photomicrographs also show that the nature of the cracks which developed within each coating was different. The thermal shock testing caused a mud-cracking pattern to develop within the chromia-silica coating (region labeled M in Figure 3.5.5). Also observed on these specimens was cracking within the cast iron substrate (region labeled A in Figure 3.5.5). The cracks observed within the high carbon iron-molybdenum coating were radial cracks (region labeled R in Figure 3.5.6). In addition, no excess oxidation was observed on any of the plasma sprayed coated specimens after completion of the thermal shock testing.

The cracking observed within these plasma spray coatings after the thermal shock testing is not considered a problem because plasma sprayed piston ring coatings used in current production diesel engines exhibit similar crack patterns and these cracks are not detrimental to the friction and wear properties of these coatings.

3.6 Additional Glassy Phase Development

The initial pin-on-disk friction and wear testing of the cast iron porcelain enamel (CIPE) composition revealed that this composition had high friction values (above 0.3) and poor wear characteristics when run against either a metallic or ceramic counterface at temperature. Hard particle additions of zirconia, alumina, or chromia did not improve the wear properties of the CIPE. These hard particle additions were unsuccessful because they increased the coefficient of friction of the CIPE to above 0.4. Increasing the coefficient of friction increased the CIPE's wear rate when run against either counterface.

These initial results illustrated that to obtain adequate wear properties, the coefficient of friction of the base CIPE composition must first be reduced to below 0.1. Then hard particles can be added, if required, to improve the wear properties. Incorporation of solid lubricants into the base CIPE composition should reduce the coefficient of friction. Using published friction and wear data as a guide, the following solid lubricants were selected to be incorporated into the CIPE: boron nitride, calcium fluoride, barium fluoride, silver flakes, and cesium oxythiomolybdate.

A potassium silicate composition was also selected to be evaluated as a potential wear resistant cylinder liner coating. The potassium silicate was selected because it can also be applied using slurry spray techniques and its curing temperature is below 400 C. Typical firing temperatures for the CIPE composition are 700 C.

In general, the additional glassy phase development program consisted of first developing robotic slurry spray application techniques and a drying and firing cycle for each composition. Next, the compatibility of the solid lubricant with CIPE and potassium silicate compositions was determined. Finally, the friction and wear properties of the CIPE and potassium silicate compositions containing the solid lubricant additions were determined using the pin-on-disk friction and wear test.

Robotic slurry spray parameters were determined for the potassium silicate and CIPE compositions to cast iron substrates. Control of the coating viscosity and solids content were important for obtaining smooth, uniform, wet coatings which can be applied to cast iron substrates.

Next, attempts were made to determine a drying and firing cycle for each coating. A drying and firing cycle was developed for the CIPE, but a drying and firing cycle for the potassium silicate could not be developed. Problems encountered during the drying of the potassium silicate included crazing and blistering. Drying and firing cycles evaluated to reduce the tendency of the potassium silicate to craze or blister included slow and/or fast heating rates, long and short soaks at various temperatures, drying with and without humidity, air drying without any external heat, and slurry spraying the potassium silicate on warm (above 100C) substrates. All these cycles were unsuccessful. For this reason, it was decided to drop this coating composition from the program.

The chemical and physical stability of the solid lubricants and the CIPE was determined by mixing the CIPE with five and 10 weight percent additions of each solid lubricant and

FIGURE 3.5.5
CHROMIA-SILICA COATED FALEX SPECIMENS
AFTER THERMAL SHOCK TESTING

MAGNIFICATION 3.5X

FIGURE 3.5.6
HIGH CARBON IRON-MOLYBDENUM COATED
FALEX SPECIMENS AFTER THERMAL SHOCK TESTING

MAGNIFICATION 3.5X

MAGNIFICATION 30X

recording the physical appearance and viscosity of each CIPE, solid lubricant mixture at regular time intervals over a thirty day period (a sudden increase or decrease in viscosity and or a change in color or physical appearance would indicate a chemical reaction occurring.). No adverse chemical reactions were observed between any of the solid lubricants and the CIPE. Then combinations of one or more of the solid lubricants were blended with the CIPE and the stability of these mixtures evaluated. Again no adverse chemical reactions were observed.

Next, the CIPE compositions containing each of the solid lubricants were slurry sprayed onto cast iron substrates and fired. Chemical interactions between the CIPE and the solid lubricants during the drying and firing cycle (i.e., some of the solid lubricants acted as a flux) required that a different firing schedule be determined for each CIPE plus solid lubricant composition. Once the proper firing cycle was determined, the friction and wear properties of each composition were characterized by pin-on-disk friction and wear testing running using a ceramic counterface at 350 C, lubricated.

The addition of the 2.5 weight percent silver flake did not reduce the coefficient of friction of the CIPE. However, increasing the silver additions to 5 and 10 weight percent reduced the coefficient of friction of the CIPE from above 0.35 to .31 and .26, respectively. Although the silver flake additions did reduce the coefficient of friction, this CIPE composition did not meet the wear goal of this program because a 2.5 mm wide wear scar developed on these coatings after less than 120 minutes of testing. Examination of the specimens after the friction and wear testing showed that the reduction in coefficient of friction occurred because the solid lubricants were smeared across the wear track. This examination also revealed that the coating crushed and then spalled under the compressive load of the ceramic pin. Thus, the low compressive strength of this coating was the reason for the poor wear rates.

Coefficient of friction values of 0.26 were obtained with a five weight percent BaF_2 addition. Reducing the friction coefficient again did not produce a coating which met the wear goals of this program because of the low compressive strength of the base enamel composition. The coating crushed and spalled under the compressive loading of the ceramic counterface during the pin-on-disk testing and a 2.5 mm wide wear scar developed within the enamel after only 120 minutes of testing. Increasing the BaF_2 content to 10 weight percent increased the coefficient of friction to 0.36. Again, coating, crushing and spallation occurred during testing and the test was terminated after 120 minutes because a 2.5mm wear scar developed.

Brown nitride (BN) additions at either a 2.5 or 5 weight percent, did not reduce the coefficient of friction of the CIPE. These compositions developed 2.5 mm wear scars running against the zirconia counterface after less than 5 minutes of testing. Again the wear mechanism was first crushing and then spalling of the coating under the compressive loading of the ceramic counterface.

The cesium oxythiomolybdate solid lubricant additions produced CIPE coatings with coefficient of friction values of 0.09 at the start of the pin-on-disk testing. However, once the zirconia counterface wore through the top surface of the CIPE, the coefficient of friction rapidly increased to above 0.36. Thus, while these compositions had acceptable wear rates at the start of the test, the wear rates increased dramatically once the zirconia counterface wore through the top surface of the coating and crushing of the coating began. On these specimens a 2.5 mm wide wear scar developed after only 30 minutes of pin-on-disk testing.

Combinations of the solid lubricants additions to the CIPE were evaluated next. These combinations included:

1. CIPE plus 5 percent silver flake and 2.5 percent BaF,
2. CIPE plus 5 percent silver flake and 5 percent BaF,
3. CIPE plus 10 percent silver flake and 2.5 percent BaF,
4. CIPE plus 10 percent silver flake and 5 percent BaF,
5. CIPE plus 5 percent silver flake, 5 percent BaF, and 5 percent CaF.

Smooth, uniform coatings with glossy physical appearances were obtained from all these CIPE, plus solid lubricant compositions. However, the pin-on-disk friction and wear test results were disappointing. Running against a zirconia counterface, a 2.5 mm wide wear scar developed on each CIPE plus solid lubricant composition, irrespective of the solid lubricant type or percentage, after less than 5 minutes of testing.

Figure 3.6.1 shows the surface after testing of three regions from the same CIPE plus 5 percent silver flake and 2.5 percent BaF solid lubricant coated specimen. Very little wear is apparent on the surface contained in Figure 3.6.1A. Extensive spalling of the CIPE was observed in Figure 3.6.1C (area C). Also observed in Figure 3.6.1C, were many bubbles (area D) in the CIPE microstructure. No bubbles were observed in the region which showed very little wear (Figure 3.6.1A). A moderate amount of spallation can be observed on the surface shown in Figure 3.6.1B. In this photomicrograph, the spallation, labeled B, initiated in areas where bubbles were on or close to the surface. Thus, the reason for the poor wear characteristics of the CIPE was the large amount of bubbles in the CIPE. The bubbles reduced the compressive strength of the CIPE, and for this reason, the CIP crushed under the load of the ceramic counterface during testing.

FIGURE 3.6.1
CIPE SURFACES AFTER PIN-ON-DISK TESTING
(MAGNIFICATION 25X)

**A. REGION WITH VERY
LITTLE WEAR**

B. MODERATE WEAR REGION

C. SEVERE WEAR REGION

FIGURE 3.6.2
CROSS-SECTIONAL OPTICAL PHOTOMICROGRAPH
OF CIPE PLUS SOLID LUBRICANT COMPOSITION
APPLIED TO CAST IRON SUBSTRATE

MAGNIFICATION 200X

CIPE

**CAST
IRON**

FIGURE 3.6.3
EFFECT OF HEATING RATE ON CIPE PLUS
SOLID LUBRICANT MICROSTRUCTURE
(MAGNIFICATION 200X)

A. SLOW HEATING RATE

B. FAST HEATING RATE

C. FAST HEATING RATE
WITH LONG SOAK TIME

A cross-sectional optical photomicrograph of a CIPE plus solid lubricant coating applied to a cast iron substrate is contained in Figure 3.6.2. This photomicrograph shows a large amount of bubbles in the CIPE (dark circles labeled F). The bubbles range in size and are located across the entire width of the coating. This photomicrograph also shows that the solid lubricant additions (white particles labeled A) are also uniformly distributed throughout the CIPE.

After the mechanism for the poor compressive strength of the CIPE was identified, a program was initiated to determine if the number and size of the bubbles could be reduced by altering the CIPE's firing cycle. The firing cycles evaluated consisted of very fast heating rates, i.e., placing the specimen in a hot furnace, and very slow heating rates, 2 C/minute, in addition to long and short soak times at temperature.

The results of these firing studies are illustrated in Figure 3.6.3. Using a slower heating rate, Figure 3.6.3A, reduced the size of the bubbles. However, the number of bubbles contained in the microstructure increased. Large bubbles were characteristic of the faster heating rate. These large bubbles, Figure 3.6.3B, were concentrated at the CIPE, cast iron interface. Increasing the soak time did not eliminate the bubbles, Figure 3.6.3C. Instead, the longer soak times caused the solid lubricant particles to coalesce into larger particles located at either the top of the CIPE or at the CIPE, cast iron interface. In conclusion, none of the firing cycles evaluated resulted in a bubble-free CIPE microstructure.

4.0 Conclusions

Chemical or physical vapor deposition and plasma spray techniques can be used to apply wear resistant coatings to the outer diameter of piston rings. Wear resistant coatings can be applied to the inner diameter of cylinder liners using low temperature arc vapor deposition and plasma spray techniques.

Wear resistant coatings applied using chemical, physical, or low temperature arc vapor deposition techniques deposit coatings which conform to the topography of the surfaces to which they are applied. Thus, if these methods are used for applying coatings to surfaces which already have the desired surface finish, these coatings will not require any additional machining after application.

Falex pin-on-disk and Hohman A-6 friction and wear testing identified two wear resistant coating pairs which met the friction and wear goals of this program. The coating pairs identified were:

1. A plasma sprayed high carbon iron-molybdenum coated piston ring running against a plasma sprayed chromia-silica coated cylinder liner and,
2. A plasma sprayed high carbon iron-molybdenum coated piston ring against a low temperature arc vapor deposited chrome nitride coated cylinder liner.

Chemical, and low temperature arc vapor deposition techniques, using current processing technology, can not apply coatings thick enough to meet the existing commercial diesel engine durability requirements. To meet the current commercial diesel engine durability requirements, coatings with thicknesses greater than 15 microns are required. For this reason, additional process development is needed to learn how to apply uniform, adherent coating using either chemical or low temperature arc vapor deposition techniques.

Coefficient of friction values obtained from wear resistant coatings in sliding applications are dependent, in part, by the surface finish of the sliding surfaces. High carbon iron-molybdenum coated rub shoes with a surface finish above 40 microinches exhibited 0.4 coefficient of friction when run against a plasma sprayed chromia-silica coated disk with a surface finish of 5-10 microinches. These high coefficient of friction values increased the contact surface temperatures to well above 350 C. For this reason, this material pair had an average wear coefficient of 10^{-5} mm³/N-m. These high carbon iron-molybdenum rub shoes were reground to obtain a 5-10 microinch surface finish. The reground shoes recorded a coefficient of friction value to less than 0.1, running against the chromia-silica coated disk. The lower coefficient of friction values reduced the average wear coefficient for this material pair to less than 10^{-8} mm³/N-m.

The adhesion of coatings with different chemistries, and applied using different methods (i.e., plasma spray or chemical vapor deposition) can not be evaluated using the Revetest. Instead, a modified tensile pull adherence test should be used for evaluating the adhesion of these coatings. The tensile pull adherence test showed that adherence values of above 9 MPa are adequate for wear resistant coatings in sliding applications.

The cast iron porcelain enamel compositions evaluated in this program had good adhesion to the metallic substrates to which they were applied. However, this enamel composition running at 350 C, lubricated, against a ceramic counterface in a pin-on-disk friction and wear test had coefficient of friction values above 0.25 and very poor wear coefficients (10^{-3} mm³/N-m). Addition of hard particles to the enamel composition increased the coefficient of friction values of these compositions to above 0.3. A small decrease in coefficient of friction values was obtained, for these compositions, by the addition of silver flake or barium fluoride solid lubricants to the base enamel composition.

Bubbles, in the enamel microstructure, reduced the compressive strength of the enamel compositions and were the reason for the extremely poor average wear rates of all the enamel compositions.

5.0 Recommendations For Additional Work

The Hohman friction and wear test and the characterization studies have identified the plasma sprayed high carbon iron-molybdenum, the plasma sprayed chromia-silica as potential wear resistant piston ring coatings and the low temperature arc vapor deposited chrome nitride and plasma sprayed chromia-silica as potential wear resistant cylinder liner coatings.

All the plasma spraying in this program used an O.D. (outer diameter) plasma gun. Application of a wear resistant coating to the bore of a cylinder liner will require the use of an I.D. (inner diameter) plasma gun. Additional process optimization using the I.D. plasma spray gun is recommended. This optimization study will conclude with the plasma spraying of simulated piston rings with the high carbon iron-molybdenum and a simulated cylinder liner with the chromia-silica. The friction and wear properties of these simulated components will be further characterized for friction and wear in a reciprocating test which will closely simulate low heat rejection diesel engine operating conditions.

The wear curves developed for the chrome nitride coating showed that a 15 micron thick coating is required to meet the commercial durability requirements of a wear resistant cylinder liner coating. It is recommended that process parameters for application of a 15 micron thick low temperature arc vapor deposited chrome nitride coating to the bore of cylinder liners be developed. Upon completion of this process development, simulated cylinder liner components will be coated with and be evaluated for friction and wear running against a plasma sprayed high carbon iron-molybdenum coated piston rings in a reciprocating friction and wear test.

The glassy phase development showed that an enamel coating with bubble-free microstructure is required to prevent crushing of the enamel under the load during a friction and wear test. This program also showed that the coefficient of friction of an enamel surface can be reduced by the additions of solid lubricants to the enamel composition. It is recommended that the wear resistant enamel development be continued concentrating on enamel compositions which develop bubble-free microstructures. Then solid lubricant can be added, if required, to reduce the coefficient of friction of the enamel.

Appendix 1

Individual Pin-On-Disk Friction & Wear Results

Table 3.3.3 Pin-on-Disk Friction and Wear Test Results -- Plasma Sprayed Coatings

Pin	Test Time (min.)	Friction		Average Wear Coefficient Coating	Pin

CHROMIA-SILICA COMPOSITE					

ZrO2	120	0.09	0.15	4.28x10-7	8.93x10-8
	120	0.13	0.18	6.77x10-7	1.46x10-7
	120	0.09	0.13	2.90x10-7	9.91x10-7
	120	0.09	0.13	4.86x10-7	1.45x10-7
440 SS	120	0.04	0.10	2.97x10-7	3.49x10-7
	90	0.05	0.10	5.84x10-7	1.03x10-8
	30	0.13	0.18	1.85x10-6	6.99x10-6
	30	0.13	0.16	7.21x10-6	3.71x10-7
=====					
HIGH CARBON IRON-MOLYBDENUM COMPOSITE					

ZrO2	120	0.04	0.19	2.89x10-7	2.65x10-8
	120	0.01	0.07	8.75x10-8	8.82x10-9
	120	0.09	0.08	7.97x10-9	8.60x10-9
	120	0.07	0.13	2.33x10-7	2.33x10-8
440 SS	120	0.04	0.18	2.77x10-7	2.77x10-7
	120	0.04	0.11	4.20x10-8	4.20x10-7
	120	0.02	0.08	2.35x10-7	2.35x10-7
=====					
PS212					

ZrO2	120	0.04	0.13	3.01x10-7	4.82x10-8
	120	0.09	0.15	1.09x10-7	5.00x10-8
440 SS	30	0.09	0.20	2.42x10-6	2.64x10-6
	60	0.11	0.26	6.06x10-7	5.60x10-6
=====					
HIGH COBALT-CHROMIUM WITH TRIBALLOY INTERMETALLIC MATERIALS					

ZrO2	120	0.18	0.33	2.52x10-7	4.07x10-7
	120	0.18	0.22	2.72x10-6	2.57x10-7
440 SS	120	0.18	0.33	2.52x10-7	4.07x10-7
=====					
NICKEL, CHROMIUM, MOLYBDENUM BLEND					

ZrO2	120	0.07	0.13	3.78x10-8	1.98x10-8
	120	0.09	0.13	3.65x10-8	1.98x10-8
440 SS	120	0.15	0.26	4.07x10-7	5.46x10-7
	60	0.20	0.11	3.51x10-7	1.99x10-6

Chromia-silica, high carbon iron-molybdenum, and PS 212 evaluated further because they met the program goals with respect to friction coefficient and pin and disk wear rates.

High cobalt-chromium with triballoy intermediate and high nickel, chromium coatings were dropped because of high coefficient friction and either pin or disk wear coefficients.

Table 3.3.3 (Cont'd)
Pin-On-Disk Friction & Wear Test Results

Pin	Test Time (min.)	Friction		Average Wear Coefficient (mm ³ /N-m)	
		Min.	Max.	Coating	Pin

GRADED ZIRCONIA					

ZrO2	120	0.14	0.20	1.63x10-6	1.96x10-7
	120	0.13	0.18	2.13x10-6	7.11x10-8
440SS	60	0.15	0.24	9.16x10-7	1.33x10-6
	120	0.15	0.22	4.81x10-6	7.64x10-8
=====					
ZIRCONIA, TITANIA, YTTRIA COMPOSITE					

ZrO2	120	0.13	0.20	1.28x10-7	4.11x10-8
	112	0.18	0.37	4.29x10-7	1.99x10-7
=====					
HIGH MOLYBDENUM, NICKEL, CHROMIUM BLEND					

ZrO2	120	0.09	0.18	6.02x10-8	3.33x10-7
	120	0.04	0.22	1.79x10-8	2.47x10-7
	120	0.09	0.24	4.34x10-8	2.89x10-7
	120	0.09	0.19	1.06x10-8	4.43x10-7
440 SS	30	0.09	0.20	2.64x10-8	1.21x10-6
	90	0.11	0.29	9.29x10-8	1.12x10-6
	60	0.09	0.22	3.16x10-7	1.40x10-6
Al2O3	116	0.09	0.24	5.61x10-8	3.88x10-7
=====					
NICKEL-CHROMIUM ALLOY					

ZrO2	120	0.09	0.15	3.60x10-7	6.16x10-9
	120	0.09	0.15	8.82x10-7	1.19x10-8
	120	0.12	0.16	1.66x10-6	1.08x10-8
	120	0.09	0.18	3.42x10-7	2.52x10-9
440 SS	120	0.11	0.15	2.64x10-8	1.21x10-6
	120	0.11	0.20	9.29x10-8	1.12x10-6
	60	0.09	0.22	3.16x10-7	1.40x10-6
Al2O3	116	0.09	0.24	5.61x10-8	3.88x10-7
=====					

Table 3.3.4.
Pin-On-Disk Friction & Wear Test Results--Chemical, Physical Deposited Coatings

Pin	Test Time (min.)	Friction		Average Wear Coefficient (mm ³ /N-m)	
		Min.	Max.	Coating	Pin
-----LOW TEMPERATURE ARC VAPOR DEPOSITED (LTAVD) CHROME NITRIDE-----					
ZrO ₂	120	0.10	0.19	1.87x10 ⁻⁹	3.21x10 ⁻⁸
	120	0.08	0.19	6.05x10 ⁻⁸	2.48x10 ⁻⁸
440 SS	30	0.18	0.33	8.36x10 ⁻⁷	5.80x10 ⁻⁶
	120	0.10	0.11	6.58x10 ⁻⁷	1.63x10 ⁻⁸
=====					
-----LAYERED CHEMICAL DEPOSITED TITANIUM NITRIDE/ TITANIUM CARBIDE-----					
ZrO ₂	120	0.10	0.13	6.06x10 ⁻⁹	9.16x10 ⁻⁹
	120	0.10	0.18	1.70x10 ⁻⁸	6.08x10 ⁻⁹
440 SS	30	0.18	0.22	1.09x10 ⁻⁶	5.53x10 ⁻⁶
Al ₂ O ₃	120	0.08	0.11	4.45x10 ⁻⁸	6.76x10 ⁻⁹
=====					
-----MID-TEMPERATURE CHEMICAL VAPOR DEPOSITED TITANIUM CARBONITRIDE-----					
ZrO ₂	120	0.09	0.11	4.25x10 ⁻⁸	6.01x10 ⁻⁸
	120	0.07	0.10	3.72x10 ⁻⁸	2.55x10 ⁻⁸
440 SS	30	0.24	0.24	2.53x10 ⁻⁶	1.20x10 ⁻⁵
Al ₂ O ₃	120	0.08	0.15	3.14x10 ⁻⁸	6.83x10 ⁻⁹
=====					
-----CHEMICAL VAPOR DEPOSITED CHROME CARBIDE-----					
ZrO ₂	60	0.09	0.18	4.73x10 ⁻⁷	1.38x10 ⁻⁸
440 SS	30	0.24	0.31	7.68x10 ⁻⁶	3.89x10 ⁻⁵
Al ₂ O ₃	120	0.11	0.22	9.12x10 ⁻⁷	1.32x10 ⁻⁸
Si ₃ N ₄	48	0.13	0.59	6.30x10 ⁻⁷	1.98x10 ⁻⁷
=====					

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