Low-friction Hard Coatings

Principal Investigators: A. Erdemir and O. L. Eryilmaz
Argonne National Laboratory, Energy Technology Division, Argonne, IL 60439-USA

The submitted manuscript has been created by the University of Chicago as Operator of Argonne National Laboratory ("Argonne") under Contract No. . DE-AC02-06CH11357 with the U.S. Department of Energy. The U.S. Government retains for itself, and others acting on its behalf, a paid-up, nonexclusive, irrevocable worldwide license in said article to reproduce, prepare derivative works, distribute copies to the public, and perform publicly and display publicly, by or on behalf of the Government.

December 2011

DOE EERE FY 2011 Progress Report

*Work supported by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Freedom Car and Vehicle Technologies Program, under Contract No. DE-AC02-06CH11357.

Agreement 13721 – Low-friction Hard Coatings

Principal Investigators: A. Erdemir and O. L. Eryilmaz

Argonne National Laboratory
9700 S. Cass Avenue, Argonne, IL 60439-4838
(630) 252-6571; fax: (630) 252-5568; e-mail:Erdemir@anl.gov; Eryilmaz@anl.gov

DOE Technology Manager: Jerry L. Gibbs
(202) 586-1182; fax: (202) 586-1600; e-mail: jerry.gibbs@ee.doe.gov

Contractor: UChicago Argonne LLC
Contract No.: DE AC03 06CH11357

Objective

- Design, develop, and implement super-hard and low-friction coatings to increase the durability, fuel economy, and environmental compatibility of future engine systems.
- Characterize and verify coating performance through bench-top, fired-engine, and field studies.
- Reduce emissions through reduction of fuel consumption as well as sulfur and phosphorous from engine oils.
- Demonstrate large-scale production of optimized coatings in a commercial-scale deposition system and transfer optimized technology to industry.

Approach

- Optimize coating microstructure, chemistry, thickness and surface finish that were proven to be very important for mechanical and tribological properties of superhard and low-friction coatings.
- Develop reliable deposition protocols for large-scale manufacturing of MoN-Cu coatings.
    - Confirm superior bonding and surface smoothness.
    - Confirm super-hardness and low friction.
    - Confirm extreme resistance to wear and scuffing under prototypical conditions.
- Demonstrate large-scale production with good reproducibility and cost-competitiveness, and verify durability and performance in actual engine applications.
- Passenger car engine tappets, fuel delivery systems, and transaxles are selected as a target applications for our coatings in FY2011.

Accomplishments

- Optimized coating-substrate bonding for extreme engine operating conditions (strong bonding was a must for differential, camshaft, and tappet applications where contact stresses were very high).

- Validated large-scale manufacturability of optimized coatings in commercial-scale deposition systems.
- Demonstrated batch-to-batch repeatability of coating quality and performance. Develop faster and more efficient ways to clean, load, unload, and inspect coated engine parts.
- Developed more effective quality control procedures and deposition protocols for large-scale manufacturing.
- Explored the possibility of manufacturing superhard coatings using high-power impulse magnetron sputtering (HIPIMS) technique and achieved much harder and more adherent coatings.
- Made progress toward new coating procedures to eliminate the need for post-deposition polishing.
- Coated several engine components (tappets, cam shafts, differential parts, rocker arms, piston rings, and piston pins) and delivered them to multiple OEMs and automotive companies for internal evaluation and engine testing.
- Demonstrated excellent performance on coated engine parts.
- Developed and implemented a special surface conditioning protocol to achieve extremely smooth surface finish with right kinds of chemistry providing very low friction and wear from the very beginning (no run-in).
- Filed a new patent application on this surface conditioning method (ANL-IN-10-008) and was granted US patent (7,846,556) on modulated composite surfaces that combine low-friction and superhard coatings with different surface textures and modulations.
- Made progress toward full-scale commercialization, licensing of technology is in the final stages.

Introduction

Higher energy efficiency, longer durability, and lower emissions are much desired attributes in future transportation systems; but, without further improvements in the surface mechanical and tribological properties of sliding, rolling, or rotating engine components, these goals will be difficult to realize. In particular, higher loads, speeds, temperatures, and other harsh operating conditions in future engines will render most traditional materials and lubricants ineffective. Accordingly, in this project, using an advanced physical vapor deposition process, we developed a variety of novel nano-composite coatings that are not only super hard (up to 67 GPa) and hence wear resistant but also super low friction (less than 0.03) when tested under boundary lubricated sliding conditions. These nano-structured and composite coatings consist of a MoN hard phase and a Cu soft phase.

The hard and soft phases in these coatings provide a very dense, equiaxed structure which is responsible for extreme hardness, toughness, as well as excellent responsiveness to the additives in lubricating oils. Specifically, they react with sulfur and phosphorous in lubricating oils during sliding and form highly protective and low-friction boundary films that prevent wear and scuffing. Some of the most effective coating compositions we have developed so far include 80-98% MoN or Mo_2N and 2-20% Cu or other soft metals (such as Zn, Ag, Sn, Sb, etc.) by weight. Systematic tribological studies of these coatings in EGR contaminated engine oils showed that they are also very effective in reducing friction and wear and preventing corrosion and abrasion in such oils. Overall, comprehensive friction, wear, and scuffing studies in our laboratory have indicated that these coatings could be extremely useful for a wide range of engine applications including those supported by the Vehicle Technologies Program of DOE. Specifically, they can reduce parasitic friction losses (hence increasing fuel economy) and wear (hence increasing component durability/reliability) in a variety of engine parts, including, piston pins and rings, camshafts, transmission and transaxle parts and tappets.

During FY 2011, we made more progress toward large-scale production, field testing, and licensing of our superhard and low-friction coating technology. Specifically, we worked very closely with Hauzer Technocoating (one of the largest company in the field of PVD coating) and Galleon International to produce these coatings on functional surfaces of numerous engine parts using production-scale deposition systems. We also worked closely with several engine companies and successfully deposited our superhard and slick coatings on their engine parts and components for performance and durability tests in simulated and actual engines.

Experimental

During FY2011, we completed all of our deposition trials, bench-top tribological testing and surface analytical studies. Specifically, we perfected the procedures for deposition of high-quality superhard coatings and post-process surface conditioning using commercial-scale deposition systems and industrially relevant polishing procedures.

Comparison of the mechanical and tribological properties of competing coatings (e.g., CrN and DLC) has also been finalized. Overall, much of the effort was directed toward component level and actual engine studies.

During our optimization studies in FY2011, a special emphasis was placed on development and implementation of an effective protocol for eliminating the surface roughness which was found to be detrimental to the friction and wear resistance of most coated engine parts. Working very closely with industrial partners, we developed and optimized a post-deposition surface conditioning and polishing method which in the end provided very consistent and smooth surface finish which in turn provided the level of improvements that we have been looking for in friction and wear performance. These novel procedures have been filed as a new invention and then as a patent.

Due to much reduced sulfur in fuels and higher compression and temperature regimes in diesel engines, and to adaptation of very low viscosity (like SAE 0W20) grade oils for higher fuel efficiency, engine companies have been complaining about increased wear in piston rings/liners and fuel injector systems. During FY2011, we developed a special protocol for the deposition of our coatings on curved piston ring and fuel injector plunger surfaces. We also employed post-deposition polishing of their surfaces to achieve very smooth surface finishes. Specifically, we stacked them up in a special holder and coated only the rubbing surfaces with 3 to 5 micrometer thick superhard coatings. With a special polishing machine, these coated surfaces were polished effectively to remove any surface roughness. Several batches of coated rings were delivered to an industrial partner for their internal evaluation and engine testing. We also received some of them for our internal evaluation and quality control work. Our evaluations showed that during the post-deposition polishing process, most of the surface irregularities were effectively removed and the remaining coating was very dense, smooth and combined all the desirable qualities for good performance in actual tests. In a few in-house trials, these coatings showed excellent adhesion and high hardness values of more than 2400 Vickers.

Results

Those highly optimized superhard coatings for engine studies were applied on a wide range of components (i.e., piston rings, tappets, piston pins, camshafts, and differential parts) using a production-scale deposition unit at Hauzer Technocoating (our coating partner). Most of the coated parts passed the initial screening tests and all of them performed extremely well in the shorter duration motored/fired engine studies. A few of them are currently undergoing long-duration engine tests in our engine OEM partners' laboratories. Initial findings suggested that these optimized coatings were extremely resistant to wear and scuffing and provided up to 90% reduction in friction (especially tappets).

A variety of tappets coated with superhard coating provided much better wear performance than DLC- and CrN-coated tappets in long-duration tests. Frictional behavior of superhard coating was impressive up to 80°C but slightly inferior to CrN coating at 120°C; CrN suffered

significant wear, while superhard coated surface showed no evidence of wear.

When tested in a differential of a full-size pickup truck under severe condition, superhard coating worked better than any other coatings tested before. Without our coating, the parts in the differential broke, causing the discs to spin at very high and uncontrollable speeds. It would eventually scuff/weld to bottom piece (see picture on right) due to high heat in addition to high stress. The superhard coated part showed no evidence of wear and protected sliding surfaces against scuffing as shown in Figure 1.

Figure 1. Condition of uncoated (bottom) and superhard coated diferential parts of a full-size pickup track. Uncoated surface scuffed badly, while superhard coated part was still in pristine condition.

In another actual high-speed hot engine tests, superhard coating prevented wear of piston rings and liners, while severe wear observed after tests on uncoated or CrN-coated rings and liners. Friction reduction and fuel economy benefits of coated rings are currently being evaluated in long-duration fired engine tests. Based on such very impressive test results from actual engines, discussions to license the technology for large-scale applications have reached the point where an actual agreement can be finalized very shortly.

Our coating trials with a new HIPIMS source have also continued in FY2011 and very impressive improvements in coating microstructure, surface finish and adhesion were achieved. With further optimization, it looks that post-deposition polishing of coated parts may be eliminated with the use of HIPIMS. Due to very high ionization efficiency and intermixing, the bonding of deposited coatings to substrates were much higher. When tested in a Daimler Benz Rockwell adhesion test machine, these coatings would result in a #1rating out of 7 (#1 representing the strongest bonding).

Licensing talks with outside collaborators are in the final stages and we expect this technology to be fully licensed by the end of 2011.

Conclusions

During FY2011, we have made great strides toward scaling up and commercializing our superhard and low-friction coating technology. Its effectiveness in reducing friction and wear and enhancing resistance to scuffing has been proven under actual engine operating conditions. Our coating partners have also adopted our technology and successfully deposited the same quality coatings in their deposition systems as we have been doing at Argonne. New field tests by several engine company partners further confirmed the effectiveness and very impressive tribological properties of our coatings under harsh conditions. Technology transfer and licensing activities are in the final stages and we

expect to see some industrial applications very shortly.

Patents and Publications

During FY2011, a new patent application has been filed, and we published and/or presented several new papers on the work that was performed under this project as listed below.

Patent Application

1. A. Erdemir, O. L. Eryilmaz, M. Urgen, and K. Kazmanli, "Method to Produce Catalytically Active Nanocomposite Coatings".

Presentations

1. Eryilmaz, O. L. Kim, J. H. Erdemir, A. Urgen, M., "Friction and wear behavior of MoN-Cu nanocomposite coatings under lubricated conditions", presented at the International Conference on Metallurgical Coatings and Thin Films (ICMCTF 2011); San Diego, CA; May 2 - 6, 2011.
2. Erdemir, A., "Advances in Surface Engineering for Demanding Tribological Applications", (Plenary Lecture), 1st International Surface Treatment Symposium, Istanbul Turkey, June 14-18, 2011.