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13. ABSTRACT *(Maximum 200 Words)*

A program of research has been conducted into the fundamentals of vapor phase lubrication for use in high temperature applications. The focus of the effort has been on the reaction mechanisms by which phosphates and phosphites decompose on metal surfaces to form thin lubricating films of polyphosphate glass. The results have explained the origins of the superior performance of aryl phosphates over alkylphosphates as vapor phase lubricants. In addition experiments have been conducted to probe the fundamentals of friction between single crystalline surfaces. These have revealed that friction anisotropy between crystalline metal surfaces is due to anisotropy in the work of bulk plastic deformation that occurs during sliding. They have also revealed effects of adsorbate layering at metal surfaces during sliding. Finally, study of the frictional properties of quasicrystals has revealed the role of surface oxides films in lowering the friction between quasicrystalline surfaces.

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AFOSR – Final Technical Report
Fundamentals of High Temperature Tribology

Grant No. AFOSR-F49620-98-1-0218
Duration - Feb. 1, 1998 to Nov. 30, 2000

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Pittsburgh, PA 15213

1. OBJECTIVE

This is the final report for a research program entitled "Fundamentals of High Temperature Tribology" which has explored some of the basic scientific issues associated with lubrication at extremely high temperatures. The principle effort has been directed towards understanding the mechanism of vapor phase lubrication by aryl phosphates. In addition there has been extensive effort aimed at understanding some of the fundamentals of friction between well defined surfaces of single crystalline metals and between the surface of quasicrystalline alloys.

2. TECHNICAL PROGRESS

2.1 Surface Chemistry of Vapor Lubricants on Metals

Vapor phase lubricants are used for extremely high temperature applications such as gas turbine engines. Rather than relying on liquid lubrication this lubrication scheme involves the introduction of vapor phase lubricant chemicals into hot gas stream flowing through the engine. The chemicals react on the surfaces of engine components to form thin lubricating films. The most commonly studied vapor phase lubricant is tricresylphosphate (TCP), $(CH_3(C_6H_4O))_3P{=}O$. This reacts on the surfaces of steel bearings to produce a film of solid lubricant that is believed to be composed of polyphosphate glass containing some carbon. The details of the reaction mechanism and the composition of the lubricant film are not well known but we have been able to make significant progress on these problems.

As a model compound for the study of vapor lubricant surface chemistry we originally began this investigation by studying trimethylphosphite (TMP), $(CH_3O)_3P$, the simplest possible organophosphorus compound containing the important P-O-C linkages that must decompose during the reactions of tricresylphosphate with metal surfaces. We have addressed the question of whether it is the P-O or the C-O bonds which are cleaved most readily. We have completed our studies on the Cu(111) and the Ni(111) surfaces. In addition there has been a study elsewhere on the Fe(110) surface. The uniform conclusion of this work is that the P-O bonds break first to produce a methoxy (CH_3O) species.

$$(CH_3O)_3P + M \rightarrow 3\ CH_3O\text{-}M + P\text{-}M\,.$$

Figure 1 illustrates temperature programmed reaction spectra (TPRS) that reveal the TMP decomposition mechanism on the Cu(111) surface. During heating methoxy groups produced by methanol adsorption are known to react on Cu(111) by β-hydride elimination to produce formaldehyde at a temperature of 405K. When TMP is adsorbed on the oxidized Cu(111) surface formaldehyde is observed to desorb also at 405K. The fact that the same product desorbs at the same temperature is very strong evidence that methoxy is the intermediate in TMP decomposition. X-ray photoemission spectroscopy support this conclusion and similar observation have been made on the Ni(111) and Fe(110) surfaces. One of the interesting features of these results on the Cu(111) surface is that the presence of oxygen activates the surface for the decomposition of TMP. This observation is quite similar to that made with TCP in that oxidized iron was found to be more reactive towards TCP decomposition than iron. The net result of our work suggests that the decomposition of the TMP is by initial dissociation of the P-O bonds to produce adsorbed methoxy groups.

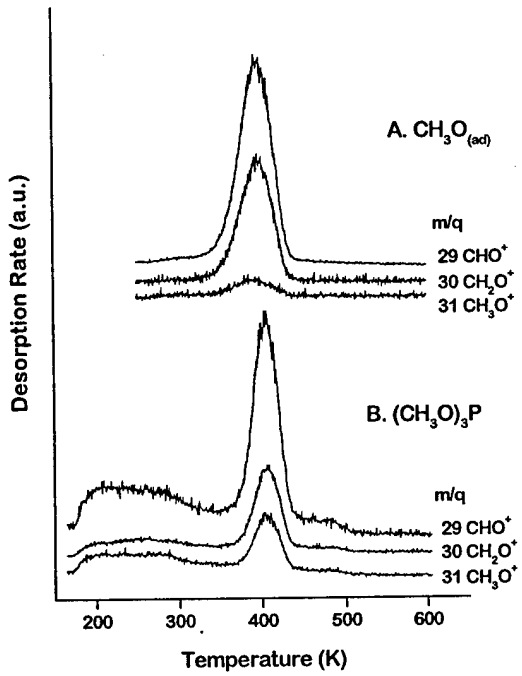

Fig. 1. TPRS of methoxy and TMP on the oxidized Cu(111) surface. The TMP decomposes to methoxy which then reacts to desorb as formaldehyde. Both desorption peaks occur at the same temperature.

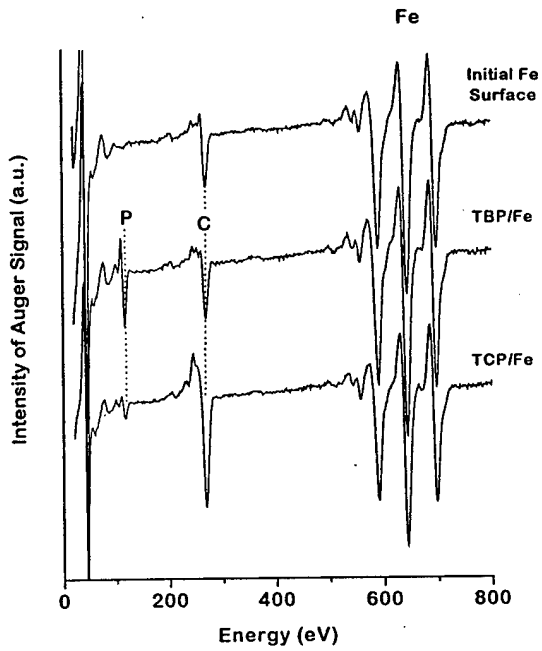

Fig. 2. Auger spectra of the an Fe foil that has been cleaned of contaminants other than some residual carbon. The lower two spectra are the same Fe foil surface after exposure to monolayers of TBP or TCP and subsequent heating to 800K. Clearly the decomposition of TCP results in the deposition of much greater amounts of carbon onto the surface.

The most recent work has progressed to the direct study of the surface chemistry of TCP itself and tributylphosphate (TBP), $[(CH_3(CH_2)_3O)_3P{=}O]$ on an Fe foil. These have allowed us to begin to explore the difference in the surface chemistry of the *alkyl* and the *aryl* phosphates. TCP and TBP are much more complex molecules than TMP and thus there are more reaction paths that are possible during their decomposition to form lubricating films. It appears from initial results that in these phosphates it is the C-O bonds that cleave on the clean Fe foil to produce adsorbed alkyl and aryl groups.

$$(RO)_3P{=}O + M \rightarrow 3\ R\text{-}M + P_xO_y$$

This is an interesting difference between the phosphites and phosphates. There is some evidence that on the oxidized surface it is the P-O bonds that break in the phosphates also but this requires further investigation. The final result is that the phosphates such as TCP can react to form either alkyl or alkoxy groups on surfaces. It is the subsequent decomposition of these adsorbed species that results in the deposition of graphite into the vapor lubricating films.

One of the interesting observations that has been made using the TCP an TBP on the Fe foil is that the decomposition of TCP leaves far more carbon on the Fe surface than does the decomposition of TBP. Figure 2 shows the Auger electron signal of the Fe foil surface before

and after exposure to single monolayers of TBP or TCP. The foil surface cannot be completely cleaned of carbon initially, however, it is perfectly clear that the decomposition of a monolayer of TBP deposits almost no carbon on the surface. In contrast the decomposition of TCP deposits a substantial amount of carbon. It is the difference in the ability of the alkyl and aryl phosphate's ability to deposit carbon on the surface that might account for the differences in their performance as vapor phase lubricants.

Alkyl and alkoxy groups appear to be important intermediates in VPL chemistry. The mechanism of their decomposition depends upon their structure and can give us some insight into the reasons why *aryl* and *alkyl*phosphates perform differently as vapor lubricants. Phenomenologically it has been observed that the alkylphosphates are less effective than the arylphosphates. This can be understood to result from the fact that the alkoxy groups produced by alkylphosphate decomposition can decompose by mechanisms which result in the removal of carbon from the surface. On the Cu(111) and Ni(111) surfaces we have studied alkoxy groups which illustrate these differences: cyclohexanoxy ($C_6H_{11}O$-), tertiary butanoxy ($(CH_3)_3CO$-), and phenoxy (C_6H_5O-). Each of these have been generated on the surface by the adsorption of their corresponding alcohols. Cyclohexanoxy groups have β-CH bonds which are easily cleaved on most metal surfaces and the species desorbs as cyclohexanone ($C_6H_{10}{=}O$) as seen in figure 3.

$$C_6H_{11}O_{(ad)} \rightarrow C_6H_{10}{=}O_{(ad)} + H_{(ad)}$$
$$C_6H_{10}{=}O_{(ad)} \rightarrow C_6H_{10}{=}O_{(g)}$$

The decomposition of *t*-butanoxy groups also proceeds via a mechanism which removes a substantial amount of the carbon from the surface. In *t*-butanoxy groups the C-O bond cleaves to produce a *t*-butyl group that then undergoes β-CH cleavage to desorb as isobutylene.

$$(CH_3)_3CO_{(ad)} \rightarrow (CH_3)C_{(ad)} + O_{(ad)}$$
$$(CH_3)_3C_{(ad)} \rightarrow (CH_3)_2C{=}CH_{2,(ad)} + H$$
$$(CH_3)_2C{=}CH_{2,(ad)} \rightarrow (CH_3)_2C{=}CH_{2,(g)}$$

In contrast, the phenoxy groups, or any related aryloxy group that might be produced from the decomposition of arylphosphates, have no β-CH bonds or comparable decomposition mechanisms that can remove carbon from the surface. Instead they decompose completely to adsorbed C, H, and O with some loss of carbon due to recombination of C and O to form CO at high temperatures.

Aryl and alkyl groups on metal surface exhibit similar difference in surface chemistry to the aryloxy and alkyloxy groups. Alkyl groups with β-CH bonds react by β-hydride elimination and the carbon is removed from the surface as olefins. This has been observed for the butyl groups on the Fe surface. In contrast the aryl groups such as toluyl that would be deposited on the fe surface during the decomposition of TCP decompose to deposit most of their carbon onto the surface. This has been observed for toluyl groups on the Fe surface explains the ability of TCP to deposit carbon. These experiments have provided a rational understanding of the differences in performance of the aryl and alkylphosphates as vapor phase lubricants in terms of their decomposition mechanisms and their relative abilities to deposit graphitic carbon onto surfaces.

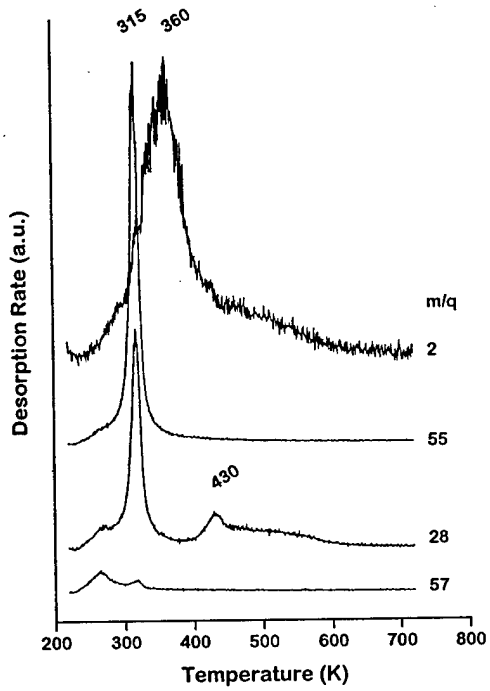

Fig. 3. TPRS of cyclohexanol on the Ni(111) surface. The sharp desorption feature at 315K for m/q = 55 and 28 is due to production and desorption of cyclohexanone which removes most of the carbon from the surface.

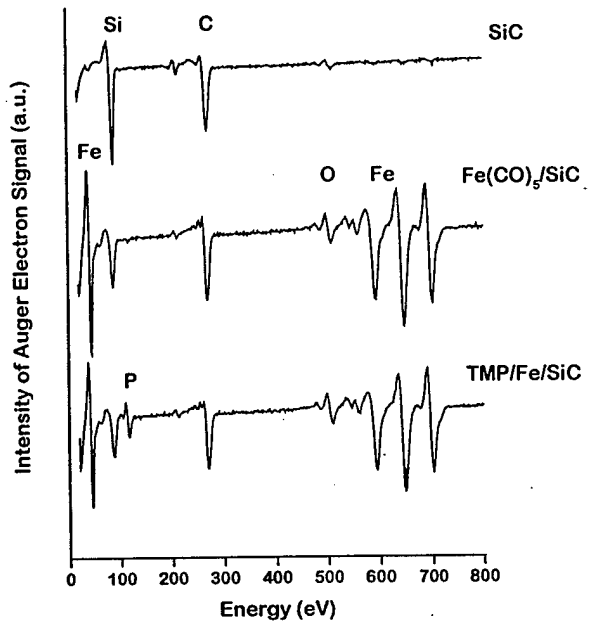

Fig. 4. Auger spectra of SiC, SiC after exposure to $Fe(CO)_5$, Fe/SiC after exposure to TMP. The middle spectrum shows the deposition of Fe onto the surface from $Fe(CO)_5$. The lower spectrum reveals the deposition of P from TMP decomposition on the SiC surface activated by Fe.

2.2 Vapor Lubrication of Ceramics

One of the most exciting results obtained during the course of the program has opened an avenue to the possible use of vapor phase lubricants with ceramic materials. Lubrication of ceramics is always more difficult than metals simply because they tend to be chemically much less reactive and because they are intended for use at extremely high temperatures. Simply exposing ceramic surfaces to vapor phase TCP does not work since the surface is not active for initiating the reactions that lead to good lubricating films. By treating the surface in a solution of iron salts it is possible to deposit a thin film of iron that will then activate the surface but this treatment cannot be performed in a continuous manner during operation. Continuous deposition of iron onto their surfaces would make VPL of ceramics feasible.

Our approach to VPL of ceramics has been to use Fe containing compounds having high vapor pressures as a sources of iron that can be deposited onto ceramic surfaces from the vapor phase continuously and concurrently with exposure to vapor phase TCP. We have chosen to use $Fe(CO)_5$ initially. Under the correct conditions it is possible to expose the surface of SiC to $Fe(CO)_5$ and observe the continuous deposition of Fe. Figure 4 shows Auger electron spectra of a SiC surface prepared simply by sputtering at 200K. This is not a clean crystalline surface and has an excess of carbon. Nonetheless exposing this surface to a flux of $Fe(CO)_5$ at a temperature of 600K and a pressure of 10^{-8} Torr clearly results in the deposition of Fe, as can be seen in subsequent Auger spectra shown in figure 4. This successful deposition of Fe is a critical and necessary step to the activation of the SiC surface for VPL.

The reactivities of the SiC and the Fe/SiC surfaces have been studied by exposing them to TMP and performing TPRS. The "nominally clean" SiC surface (without Fe) does not dissociate TMP. By contrast when TMP is adsorbed on a SiC surface that has been activated by Fe deposited from $Fe(CO)_5$, it reacts to produce CO and H_2, leaving phosphorus on the surface. The lower Auger spectrum of figure 4 reveals the presence of P on the surface following TMP decomposition. The desorption of CO and H_2 occur at the temperatures expected for TMP decomposition on Fe. This is a very exciting result because it is a clear demonstration of SiC activation by exposure to $Fe(CO)_5$ and paves the way for the proposed investigation into the VPL of ceramics.

2.3 Friction Anisotropy at Ni(100)/Ni(100) Interfaces

The mechanical properties of two crystals in contact are anisotropic in the sense that they depend upon the relative orientations of the lattices. There have been a number of suggestions that friction is high between pairs of *commensurate* periodic surfaces when they are aligned but very low when they are misaligned. This is due to the fact that the potential energy versus displacement should be corrugated when the surface lattices are aligned but smooth when they are not. Similarly the potential between two *incommensurate* surfaces should not depend on displacement suggesting that friction might be low. These ideas rest on the assumption that surfaces come into contact elastically with no deformation. While this is true in some idealized situations it is not the case for most metallic contacts under macroscopic loads. We have been able to test ideas concerning friction anisotropy with perfectly clean single crystal surfaces. We have performed measurements of the friction forces between pairs of Ni(100) surfaces as a function of the rotational misalignment between the two. These have been performed on single crystal surfaces that have been: perfectly clean, covered by ½ monolayer of sulfur in a c(2x2) array, and modified by the presence of two monolayers of ethanol. The friction coefficients as a function of misorientation angle are shown in figure 5 for all three types of interfaces. These data reveal the friction anisotropy quite clearly as a minimum at 45° misorientation. The interesting feature of this result is that it occurs at the same angle for all three surfaces. The important point is that the surface covered with the c(2x2) overlayer of sulfur has had its surface lattice rotated by 45° with respect to the Ni(100) substrates while the ethanol covered surfaces have no long range periodicity at all. The significance is that the anisotropy cannot be due to the relative orientations of the surface lattices but must be due to the relative orientations of the bulk lattices which are, of course, the same for all three surface preparations. The implication is that for surfaces in plastic contact, friction anisotropy is the result of anisotropy in the work needed to cause bulk plastic deformation during sliding rather than the relative orientations of the surface lattices. The unique feature of our experiment is the ability to measure friction between single crystalline materials in different crystallographic orientations while independently controlling the orientations and order of their surfaces.

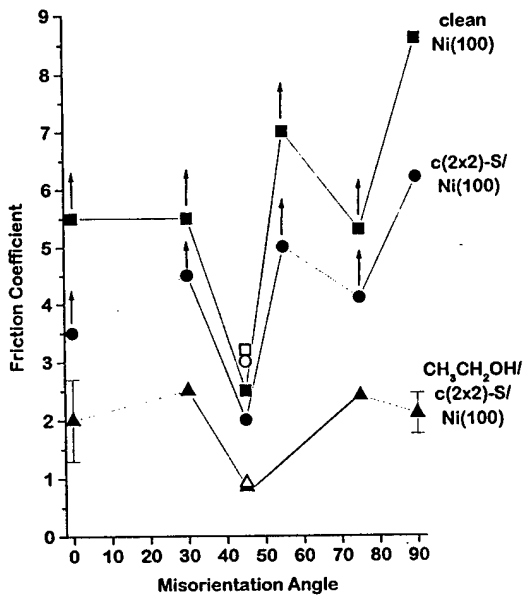

Fig. 5. Friction coefficients between Ni(100) surfaces as a function of lattice misorientation angle. The Ni(100) surfaces were: clean, modified by ½ monolayer of sulfur, or modified by 2 monolayers of ethanol. The open data points were obtained at 135° which is crystallographically identical to 45° misorientation. Data points with arrows are lower limits on μ_s.

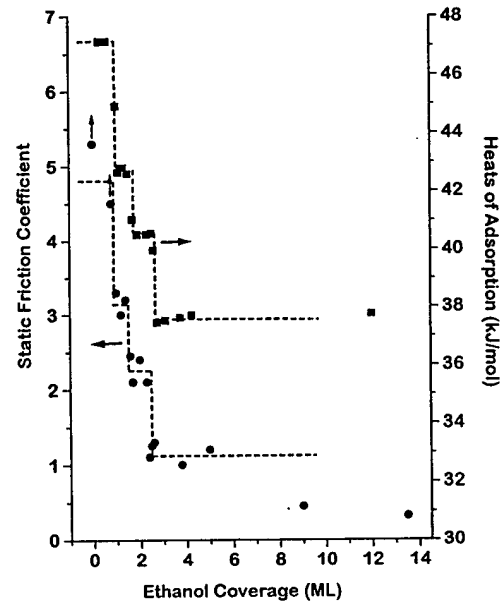

Fig. 6. Friction coefficient and heat of adsorption versus CH_3CH_2OH coverage on the c(2x2)-S/Ni(100) surface. The discontinuities in the friction coefficient appear to be correlated to the discontinuities in the heat of adsorption of ethanol. Both phenomena may be due to layering of the ethanol with increasing coverage.

2.4 Lubricant Layering at Ni(100)/Ni(100) Interfaces

The lubricating properties of thin films are influenced by their surface chemistry and their interactions with surfaces. This is difficult to study without the ability to prepare and characterize adsorbed lubricant films under conditions such as those used in our experiments. One of the interesting properties of lubricant films that has been observed with the surface forces apparatus is their ability to form discrete layers when trapped between surfaces in contact. The mechanical properties of such interfaces might then be expected to be discontinuous functions of the coverage of adsorbed lubricants.

We have studied the frictional properties of Ni(100) surface modified by ½ monolayer of sulfur and then by the adsorption of ethanol at varying coverages. The role of the sulfur is primarily to passivate the chemical activity of the surfaces and prevent the decomposition of the ethanol. We began by using thermally programmed desorption (TPD) to measure the desorption energy of the ethanol on the sulfided Ni(100) surface as a function of coverage. The results, shown in figure 6, indicate that the desorption energy is a discontinuous function of coverage suggesting that the ethanol may be forming discrete layers on the surface. We have measured the friction coefficients between two Ni(100) surfaces modified by adsorbed ethanol in the same coverage range and found evidence that friction also is a discontinuous function of coverage. Most importantly figure 6 reveals that the dependence of the friction coefficient on coverage appears to mimic that of the desorption energy. This very interesting result suggests that layering of the ethanol at the interface dictates its properties as a lubricant in much the same way as has been observed using the surface forces apparatus. The surprising aspect of our result is

that it is obtained between Ni(100) surfaces in plastic contact. The implication is that although the surfaces deform irreversibly during contact the adsorbed ethanol remains at the interface.

2.5 Quasicrystal Friction

During the latter part of this grant period a project was initiated to examine the frictional and surface chemical properties of quasicrystals. Prior work on these materials was initially funded by the DOE through Ames lab but has made use of the UHV tribometer that has been built with AFOSR support. That work has made use of $Al_{70}Pd_{21}Mn_9$ quasicrystals and their cubic approximants with composition $Al_{48}Pd_{42}Mn_{10}$. We have been able to show with those materials that the frictional properties of quasicrystalline surfaces measured in air are far different from those of truly clean quasicrystals. The presence of airborne contaminants and oxide films greatly reduces the friction of their surfaces. One of the interesting things that we have demonstrated is that the friction between perfectly clean quasicrystal surfaces of the is about half that of the approximant alloys. Most recently measurements have been made using the AlCuFeCr quasicrystal alloys prepared by TA&T Inc. These are alloy samples that have been prepared as thin sputtered films. Initial work has shown that the friction between their surfaces is dependent on temperature and shows a minimum at ~500K. At this point in time work is continuing that is aimed at understanding the surface chemical issues that dictate the frictional properties of the alloy surfaces.

3. MAJOR ACCOMPLISHMENTS

3.1 Instrumentation Development for Tribology Fundamentals

In addition to the research progress described above we have made some major improvements to our apparatus for UHV tribology. This has been possible through an equipment grant from the DURIP program. In its previous incarnation our apparatus for UHV friction measurements allowed us to control the initial properties (composition, order, and crystallographic orientation) of two single crystal surfaces prior to making measurements of friction between them. The limitations of this experiment were that it was not possible to analyze the microscopic contact regions of the surfaces after they had been separated. Our understanding of the effects of surface properties on friction was based on knowledge of the surface characteristics before they were brought into contact. Over the past year we have modified this apparatus to include a Scanning Electron and Scanning Auger microscope (SEM and SAM). This now allows us to analyze the surfaces in the microscopic contacts or wear tracks that have been produced on the surfaces during sliding. This new capability promises to greatly improve the scope of our understanding of the effects of surface characteristics on interfacial friction.

In addition with the support of another DURIP grant we are in the process of building a second UHV tribometer chamber. This will greatly accelerate the measurements of friction made under highly controlled conditions. These are very time consuming measurements and thus having a second instrument will greatly improve our productivity.

3.2 Vapor Phase Lubrication of Ceramics

During the grant period we have been able to demonstrate feasibility of a method for VPL of ceramics. The primary limitation on the VPL of ceramics is the fact that their surfaces are

chemically inert and thus do not dissociate the arylphosphates. As a means of activating the surface of SiC we have exposed it to a volatile Fe containing compound, $Fe(CO)_5$. This results in the deposition of Fe onto the SiC surface and activates it for decomposition of our model compound TMP. Successful VPL of ceramic materials represents a major milestone in the effort to develop extremely high temperature, high efficiency, and low weight engines.

4. TRANSITIONS

Seagate Technology, Inc. Research in tribological problems at the head-disk interface of magnetic storage devices is being conducted in my laboratory with support of Seagate. This work focuses on the use of some lubricants common to AF applications and has certainly benefited from the expertise derived from research efforts supported by the AFOSR. Current collaboration is through Dr. Jing Gui.

National Storage Industries Consortium (NSIC). Research is supported in the P.I.'s lab on some fundamental issues in the lubrication of hard disk surfaces. Again this makes use of some knowledge and expertise derive from AFOSR support research. The primary contact with NSIC is through Dr. Singh Bhatia.

Platinum Research Organization (PRO). I serve as a consultant to PRO in support of their program to develop lubricant formulations based on the use of fluorocarbon additives. Some instrumentation supported by AFOSR funding has been used in this endeavor. Contact is through Mr. F. Conrad Greer.

Dept. of Energy - Ames Lab I have participated in a program to study the frictional properties of some quasicrystalline alloys made at the Ames Lab. This work has made use of instrumentation built in my lab with AFOSR support. Contact is Prof. Pat. Thiel.

Kodak Corp. Research is supported in the P.I.'s lab on the oxidation of SnInSb alloys used for optical data storage. This uses the same XPS instrumentation that is used for the study of quasicrystal oxidation and many of the issues are similar to those encountered in study of quasicrystal oxidation. Contact is Dr. R. Spahn.

5. PERSONNEL

Prof. Andrew J. Gellman	10%
Dr. Daxing Ren (PD)	100%
Mr. Jeff Ko	100%
Mr. Chris Mancinelli	100%

6. PUBLICATIONS & PRESENTATIONS

6.1 Refereed Journal Articles

1. D. Ren, A.J. Gellman, "Initial Steps in the Surface Chemistry of Vapor Phase Lubrication by Organophosphorus Compounds" *Trib. Lett.* 6(3,4), (1999), 149-157

2. J.S. Ko, A.J. Gellman, C. Jenks, T. Lograsso, P.A. Thiel, "Friction Between Single Grain $Al_{70}Pd_{21}Mn_9$ Quasicrystal Surfaces" *Surf. Sci.* 423, (1999), 243-255

3. A.J. Gellman, "Transition States for Surface-Catalyzed Chemistry " *Acc. Chem. Res.* 33, (2000), 19-26

4. D. Ren, A.J. Gellman, "The Carbon Deposition Mechanism in Vapor Phase Lubrication" *Trib. Trans.* 43(3), (2000), 480-488

5. J.S. Ko, A.J. Gellman, "Frictional Anisotropy at Ni(100)/Ni(100) Interfaces" *Langmuir* 16(22), (2000), 8343-8351

6. J.S. Ko, A.J. Gellman, "The Effects of Adsorbate Layering on the Friction at Ni(100)/Ni(100) Interfaces" in press *J. Phys. Chem.*

7. D. Ren, G. Zhou, A.J. Gellman, "The Decomposition Mechanism of Trimethylphosphite on Ni(111)" *Surf. Sci.* 475(1-3), (2001), 61-72

8. D. Ren, A.J. Gellman, "Activation of the SiC Surface for Vapor Phase Lubrication by Fe Chemical Vapor Deposition from $Fe(CO)_5$" in press *Trib. Lett.*

9. A.J. Gellman, J.S. Ko, "The Current Status of Tribological Surface Science" in press *Trib. Lett.*

10. D. Ren, A.J. Gellman, "Reaction Mechanisms in Organophosphate Vapor Phase Lubrication of Metal Surfaces" in press *Trib. Int.*

6.2 Book Chapters

11. A.J. Gellman, "Tribology", *Encyclopedia of Chemical Physics and Physical Chemistry* Ch. C2.9 in press

6.3 Proceedings Article

12. A.J. Gellman, "Frictional Properties of Single Crystalline and Quasicrystalline Surfaces", *MRS Proc.* Symposium on "Tribology on the 300th Anniversary of Amonton's Law" eds. M.D. Drory, M.O. Robbins, (1999), 83-85

13. C. Mancinelli, J.S. Ko, C.J. Jenks, P.A. Thiel, A.R. Ross, T.A. Lograsso, A.J. Gellman, "Comparative Study of the Tribological and Oxidative Properties of AlPdMn Quasicrystals and Their Cubic Approximants" submitted to *MRS Proc.*

6.4 Invited Presentations

1. Univ. of California, Dept. of Chemical Eng., Berkeley, CA - Sept. 1999

2. National Research Council, Board on Chemical Science and Technology, Washington, DC - Oct. 1999

3. AVS National Meeting, Symposium on "Nanoscale Tribology and Adhesion", Seattle, WA - Oct. 1999

4. APS National Meeting, Symposium on "Friction at the Atomic Scale", Minneapolis, MN - March 2000

5. ACS National Meeting, Symposium on "Aperiodic Metals", San Francisco, CA - March 2000

6. Second San Luis Conference on Surfaces, Interfaces, and Catalysis, Mar del Plata, Argentina - April 2000

7. San Luis Summer School on Surface and Catalysis, Mar del Plata, Argentina - April 2000

8. International Conference on Metallurgical Coatings and Thin Films, Symposium on "Coating and Thin Film Analysis", San Diego, CA – April 2000

9. Sikkim International Nanotribology Symposium, Sikkim, India – May 2001