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The initial objective of acquiring instrumentation to allow microscopic tribological measurements to be made with atomic force microscopy as a function of temperature has been fully realized. This objective has been attained through the design and construction of a multi-chamber UHV surface analysis system, the design and construction of a sample transfer and manipulation hardware, and the design, installation and testing of a variable temperature, UHV, contact mode AFM. Experiments are now underway exploring the temperature dependence of fundamental tribological contacts.

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Final Technical Report

Temperature Dependent Studies of the Tribological Properties of Carbide and Nitride Hard Coatings

Air Force Contract Number
F49620-98-1-0194

Contract Dates
03/01/1998-02/29/2000

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Statement of Objectives

The original proposal requested funds to acquire a *variable temperature*, ultrahigh vacuum (UHV) atomic force microscope (AFM) and the associated surface preparation and analysis tools needed for surface characterization. Surface preparation tools were to include ion sputtering and sample annealing while surface characterization was to be carried out with low energy electron diffraction (LEED) and Auger electron spectroscopy (AES). Funds were also requested for the vacuum system (chambers, pumps, manipulator, sample transfer) to house the UHV AFM and surface analytical equipment.

The equipment was requested in order to expand the capabilities of an ongoing program in which we are studying the fundamental issues of surface chemistry and friction of hard coating materials including titanium carbide, vanadium carbide, titanium nitride. The aim of the present research program is to further the basic understanding of these materials thus enabling their use to solve tribological needs for USAF aircraft and spacecraft. This program currently involves the use of surface analysis studies including a number of techniques and the use of macroscopic pin-on-disk studies and microscopic AFM studies for tribological characterization.

The **objective** of the proposed equipment is to expand the temperature range of the tribological measurements, currently limited to room temperature. In terms of USAF applications, cryogenic temperatures are encountered in satellite mechanisms (i.e. translated detectors) while elevated temperatures are encountered in combustion engine applications. The **goal** of these studies will be to establish the how the tribological properties of hard coating contacts vary with temperature in order to more accurately predict performance properties under true application conditions. The proposed equipment will directly expand the framework of research-related education of graduate students currently working in the program.

Status of Effort

The initial objective of acquiring instrumentation to allow microscopic tribological measurements to be made with atomic force microscopy as a function of temperature has been fully realized. This objective has been attained through the design and construction of a multi-chamber UHV surface analysis system, the design and construction of a sample transfer and manipulation hardware, and the design, installation and testing of a variable temperature, UHV, contact mode AFM. The features of these components are described in the following paragraphs and figures.

The UHV surface analysis system is a multi-chamber design that allows the introduction from air of both tips and samples. The capability of low temperature experiments necessitated isolated chambers for entry, preparation, and analysis. The main analysis chamber is shown in Fig. 1 as the largest chamber on the left. The UHV STM/AFM is the smaller chamber on the right. A surface preparation chamber exists immediately behind the main analysis chamber. The custom stainless steel chambers (Huntington Laboratories) are pumped by a 240 L/sec ion and 220 L/sec turbomolecular pumps (Varian Instruments). The surface analysis capabilities of the system are centered around a reverse-view LEED Auger system (Omicron Associates), seen in the front center of the main chamber. The main chamber also houses facilities for dosing both molecular and metallic adsorbates on to the sample surface.

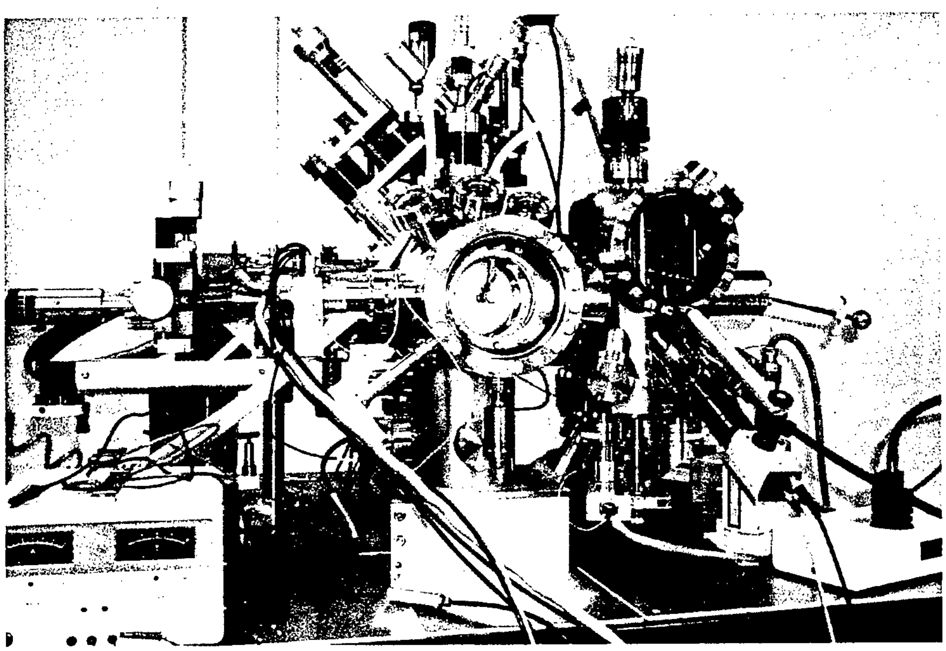

Figure 1. The UHV-STM/AFM shown to the far right is interfaced to a surface analysis system housing sample cleaning, dosing and LEED/Auger characterization facilities.

The desire to investigate metal carbide and nitride samples necessitated the design of a unique manipulator and sample preparation procedure in order to clean the samples. This need has been realized through the design(UH) and construction of a UHV sample manipulator (Thermionics Laboratories) that accepts the sample holders from the Omicron microscope. This manipulator, shown in a horizontal orientation to the lower right of Figure 2 allows heating to temperatures in excess of 1273 K and direct cooling to liquid nitrogen temperatures. The design of the microscope does not allow heating to the high temperatures required to remove oxygen from the carbide samples. The preparation chamber, shown to the left below, is isolated by a gate valve and allows sample sputtering in a location isolated from the cryogenic microscope. This chamber is also equipped with a vertically oriented secondary sample manipulator (Thermionics Laboratories) for positioning and electrical connection during the cleaning procedures. At the left of Figure 2, two horizontally and orthogonally oriented, magnetic sample transfer arms are visible which are used to introduce tips and samples to the system.

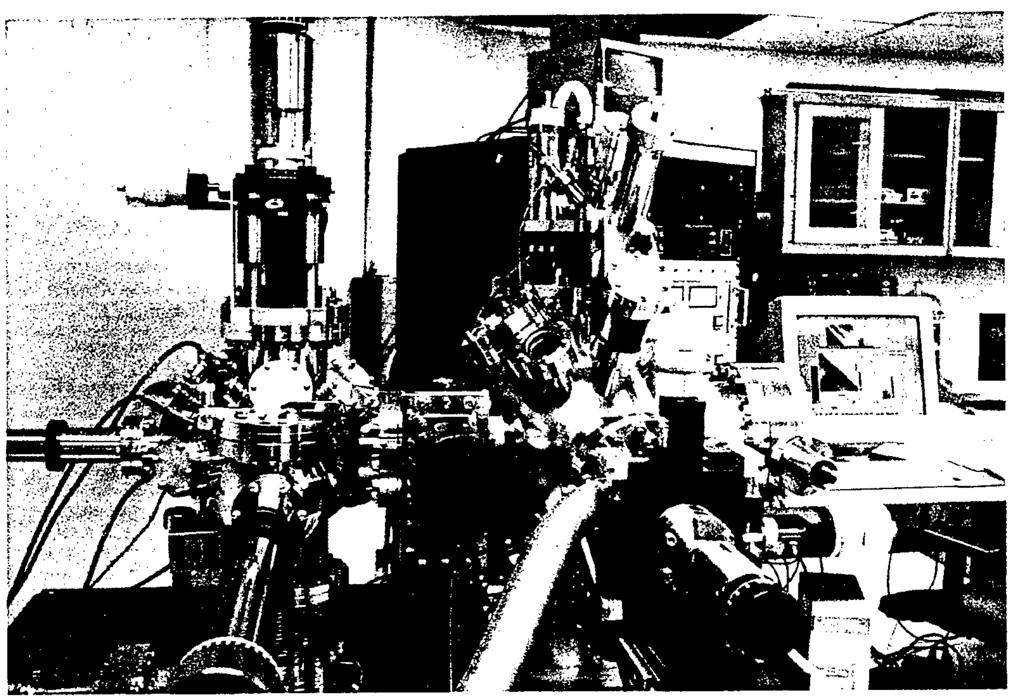

Figure 2. A side view of the system illustrates the isolated sample cleaning chamber as well as the transfer arms and load lock cell for introduction from air of tips and samples.

The UHV microscope itself is housed in a separate chamber which mounts to the chamber through an 8" flange. The unique aspects of the instrument involve a scanned contact mode AFM tip, a heated and cooled sample stage, and an optical detection scheme which involves laser light delivered from outside the chamber through a fiber optic. All electronic and heating/cooling connections are made through the lower flange of the microscope. Both samples and tips can be transferred into the microscope through the wobble stick (which protrudes to the right of Fig. 3). The microscope, including heating and cooling operations, is controlled through digital electronics and a master computer. The integrated control of sample temperature and imaging functions is critical to eliminate thermal drift and subsequent artifacts from the images and friction measuremtns.

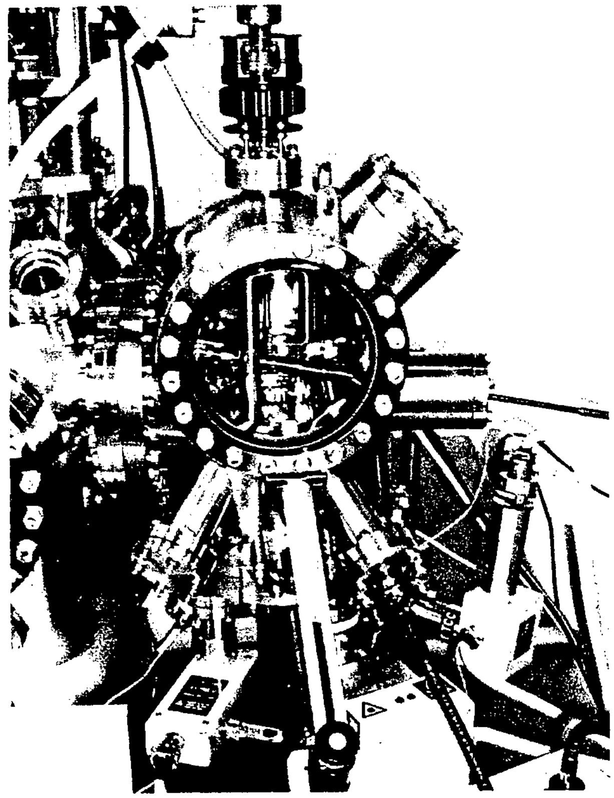

Figure 3. Variable temperature UHV-STM/AFM.

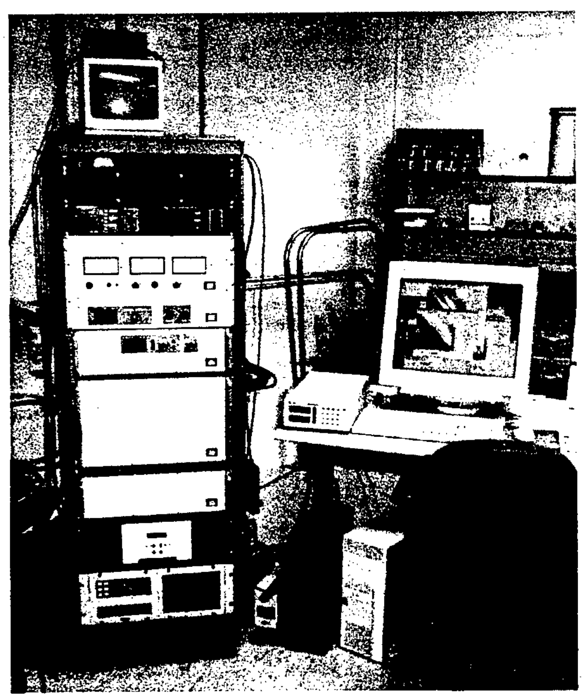

Figure 4. Computer and electronics control system of UHV microscope.

The original one year grant cycle was extended to a second year in order to complete the design and construction of the microscope. As this system represents the first of its kind in the world and required the introduction of a new sample scanning mechanism and optical detection scheme, the design and construction phase of the microscope took ~18 months. An additional 9 months were required to assemble and test the surface analysis/microscopy system. The project was somewhat hampered by the untimely departure of a senior staff member (Dr. Qidu Jiang, hired specifically to install and operate this system) after only 3 months on the project. However, at present, the microscope and vacuum system are fully installed and operational.

Initial results obtained with this instrument are displayed in Fig. 5 and demonstrate the potential importance of data derived from forthcoming studies. Here the interfacial friction measured in vacuum between a silicon nitride AFM tip and a single crystal MgO surface is plotted against the surface temperature. An approximate three-fold increase in friction is observed with a 400 degree temperature change. With associated surface analytical measurements, we are now working to establish the origin of this frictional increase.

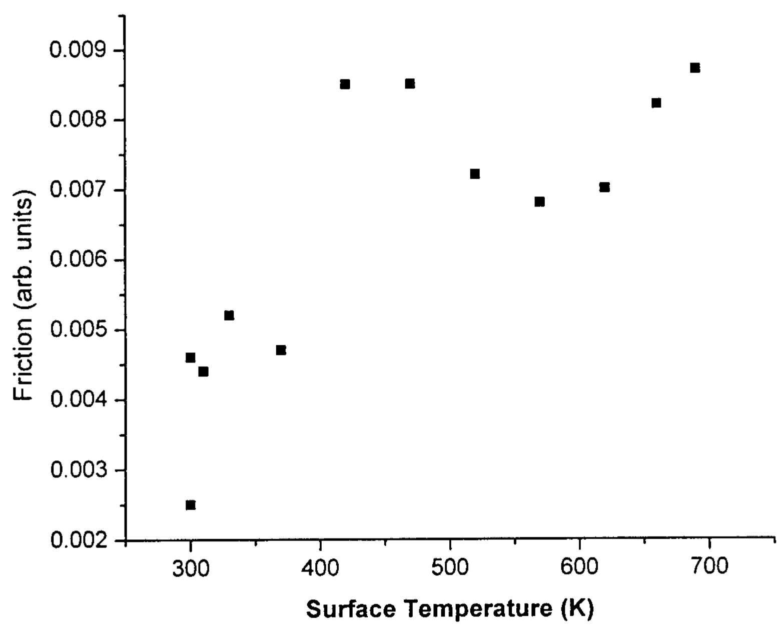

Figure 5. Interfacial friction of a Si_3N_4 probe tip and MgO(100) as function of surface temperature.

Significance to the Field

High friction and wear are problems that plague mechanical devices, often leading to degraded performance and eventual failure. To limit these deleterious phenomena, lubrication of mechanical devices is commonplace, but the lubricant type, the inclusion of boundary additives, and the need to replenish the lubricant are factors that play a significant role in the successful design and operation of a mechanical system. The USAF obviously relies upon a vast number of machines that could all benefit from a reduction in friction and wear, in turn leading to extended lifetimes, longer intervals between service, and potentially enhanced performance. We are most familiar with the needs of USAF satellite mechanical systems, specialized devices that often have very precise requirements. Such devices can represent single-point failure modes in spacecraft. Due to the inaccessibility for repair or service, these failures result in loss of mission at high strategic and financial costs. Most spacecraft mechanisms are lubricated for life during manufacture with no design for "oil changes" and often operate under unique conditions; therefore, lubricant loss and degradation, and the collection of wear debris may present significant problems during operation. The accumulation of wear debris in high precision bearings can affect performance by producing high torque and torque noise, and loss of precision movement capability through preload relief. There have been many documented and undocumented mechanical failures of satellite deployment devices, momentum control systems, scanners, actuators, and solar array drives; programs using such devices (essentially all USAF satellite systems) could potentially benefit from our work. Although the precise causes of these on-orbit failures are usually unknowable, ground testing often demonstrates that insufficient ball bearing lubrication is to blame, leading to high friction and wear. It is logical, therefore, to pursue the use of new materials that have the potential to limit wear and extend the useful life of satellite mechanisms. It is also critical to understand the temperature limitations of the new systems. The equipment provided through this contract will allow fundamental investigations of the temperature dependences of metal carbide and metal nitride coatings.

Relationship to Original Goals

The initial objective of acquiring instrumentation to allow microscopic tribological measurements to be made with atomic force microscopy as a function of temperature has been fully realized. This objective has been attained through the design and construction of a multi-chamber UHV surface analysis system, the design and construction of a sample transfer and manipulation hardware, and the design, installation and testing of a variable temperature, UHV, contact mode AFM. Experiments are now underway exploring the temperature dependence of fundamental tribological contacts.

Relevance to Air Force Mission

Advanced anti-wear materials, such as hard-coated components, are beginning to find use within Air Force, DoD, and NASA spacecraft systems. Specifically, TiC-coated bearing balls are used in the filter wheel mechanisms in the NASA/GOES spacecraft and in the reaction wheels of the NASA/AXAF (Chandra) telescope. These same materials were baselined for the reaction wheels of the SBIRS-Low FDS program. In our experience, even though the hard coatings are improving performance, several questions still remain that require a fundamental understanding of the surface chemistry and tribology of these materials and the temperature dependence of these properties. With our work, we seek to provide a fundamental perspective to enable these decisions to be made based on scientific principles.

Potential Applications to Air Force and Civilian Technology Challenges

The experiments enabled through the instrumentation fabricated under this contract will be uniquely suited to provide direct insight into the use of carbide and nitride hard coatings and the relevant temperature ranges of their successful implementation. These studies will directly benefit the Air Force in identifying (in a cost effective manner) potential temperature ranges of tribological failure, which traditionally account for significant maintenance and replacement costs, while simultaneously providing a fundamental insight into the chemical and physical origins of potential mechanical failures.

Personnel Supported by Other AFOSR Funds and Working on Project

Dr. Scott S. Perry (Associate Professor)

Luis Fernandez Torres (Graduate Student)

Publications

None to date.

Interactions/Transitions

Not applicable.

New Discoveries

Not applicable.

Honors/Awards

Scott S. Perry
University of Houston, College of Natural Science and Mathematics Teaching Excellence Award,
Spring, 2000.

Scott S. Perry (PI)
National Science Foundation CAREER Award,
April 1999.