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MAGNETRON SPUTTERED PULSED LASER DEPOSITION SCALE UP

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MATERIALS AND MANUFACTURING DIRECTORATE
AIR FORCE RESEARCH LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750

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The objective was primarily to utilize the newly developed Air Force super-hard coating process technologies in practical applications, such as turbine engine rotor components, precision shafts and ball bearings, and bearing races. This would give the Air Force the capability of practically coating real precision mechanical parts with solid lubricant films of the highest toughness. These films would be of the highest hardness and the most resilient to damage, extending the life of high cost precision aircraft components. This is driven by the fact that the primary cost in producing aircraft components is the precision machining required.

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Executive Summary: Magnetron Sputtered Pulsed Laser Deposition Scale Up

The original technical objectives of the Magnetron Sputtered Pulsed Laser Deposition Scale Up were twofold. First was the development of a Magnetron Sputtered Pulsed Laser Deposition (MSPLD) process capable of producing coated diamond like multilayer heterostructural films over large areas at low process deposition temperatures. These low deposition temperatures are critical for the coating of precision-machined mechanical parts. The objective was primarily to utilize the newly developed Air Force super hard coating process technologies in practical applications, such as turbine engine rotor components, precision shafts and ball bearings, and bearing races. This would give the Air Force the capability of practically coating real precision mechanical parts with solid lubricant films of the highest toughness. These films would be of the highest hardness and the most resilient to damage, extending the life of high cost precision aircraft components. This is driven by the fact that the primary cost in producing aircraft components is the precision machining required.

The second technical objective was to develop an automated process capable of producing large volumes of coated parts with minimal operator intervention. The production of large volumes of coated parts is necessary to reduce the intrinsic cost of each coating and take advantage of the economies of scale. The MSPLD production volume Air Force technology developed could be utilized in the case of technology transfer to aircraft component manufacturers, or other cases requiring the production of large volumes of low cost components, such as a war effort. Utilization of such advanced technology would give our soldiers an added advantage in weaponry.

From these technical objectives, a coating process was devised that would be capable of achieving these objectives in the allotted time frames and costs. The resulting system would be shipped to the Air Force Materials lab as a production ready system. The Materials lab could then utilize this process to demonstrate real technological transfer as well as produce films for volume research and quantity repeatability analysis.

As many technical objectives do, these original objectives changed during the life of the contract. The Contractor attempted to accommodate the changing Air Force demands while still meeting the original technical objectives spelled out in the contract. After meeting with the MLBT contract monitor and scientists, it was determined that the production system should also be capable of producing films at high temperature as well as low temperatures. This would permit the system to be used as a research chamber as well as a production system. To accomplish this additional technical objective, the design and incorporation of a heater stage that would fit inside the deposition process, along with changes in external hardware and software, would be necessary to incorporate this new technical objective into the original system design. The high heating of precision machined components such as bearings and races would ruin them, but components made from high temperature tolerant components, such as ceramics, would not be a problem. This would increase the capability of the deposition system so that new film material research could also be conducted on the process. There would also need to be a mode of manual operation and a way of implementing new software and hardware on the system. Fortunately, the original agreement was written such that these changes could be met without violating any of the original deliverables.

In order to accomplish these new objectives, additional funding would be required. Placing the additional funding on the contract would be accomplished by submitting a contract modification. This contract modification took an extended amount of time to accomplish, upsetting the contract schedule and fiscal panorama. Once the contract modification was settled, the work could continue on the system with the new modification incorporated into the system design, so that it would meet the objectives.

Unfortunately, the existing system design would need to be drastically changed if it was to accommodate the heater modifications. To finish the system without consideration of the heater would mean that if the heater would be purchased at a later date, the expense would be at least twice what it was if the heater was to be “designed in”. It was not possible to merely “add on” the heater as a separate purchase, but it had to be included in the design of the system. So, in order to save the costs of adding the heater later, development of the MSPLD system was halted until the contract modification was in place.

In addition, developed hardware and software was modified to account for this. No small amount of change occurred to the original proposal to accommodate the heater modification. To accommodate using the system as a research process, the software would no longer be a “turn-key” system. The instrument interface code would now be comprised of an open source code environment in LabVIEW. This open source code would facilitate the researchers in the lab, who currently use LabVIEW for all of their process control software work. Also, the instrument and control software development and maintenance would be turned over to the Air Force, so that the staff could take on improving their computer code to meet their needs. The InfoScribe data management software system would then be converted into a data server for web access, and would be accessible for data collection and archiving from LabVIEW by a set of Virtual Instrument ISX subroutines to run on a PC.

The system was developed with the heater, and performed flawlessly. The coating of multiple samples had never been done before over such a large area. It became possible to run multiple test samples rapidly, increasing the analysis of film qualities. It was possible to submit samples to several post analysis tests.

The LabVIEW software modules were incorporated and the InfoScribe data server was installed. Data archiving for the group was installed and became available to all processes through TCP and the ISTL software LabVIEW modules.

Supporting Subcontractors

PVD/Epion was the subcontractor who would develop and build the vacuum deposition hardware, heater, and chamber. The system design was conceived by Dr. Laube, and was subcontracted out to Dr. James Greer of PVD. The design is based on the concepts implemented in several thin film deposition systems. The basic design elements are based on several previously developed systems. Systems developed for high temperature superconductor research, combined with several thin film systems developed under Air Force research and development provided experience in what design elements were to be used and discarded. The general design also contains design elements utilized in production systems currently operating at Hohman Plating Co. Inc. Also implemented were two patents developed in conjunction with the Air Force past research in MSPLD. The resulting system design is a large vacuum cavity size of approximately a cubic yard, with large doors to provide access to the internals, combined with rack mount instrumentation and control logic with fault detection and interlock failsafe design.

Implementation of this effort was led by Dr. James Greer, who has successfully produced other PLD systems for the Air Force, including the HTSC system currently operating in MLPO. Dr. Greer also has decades of experience designing systems for commercial and other government research areas. Dr. Greer was able to adapt his existing design to accommodate the modifications half way through the project. Although the contract modification greatly extended the time line of this project, the ultimate savings to the government were extensive. Delivery of the MSPLD system in its original form would have eliminated the option of adding a heater later. Adding the heater would have easily doubled the system cost, requiring a new chamber and system electrical design, and not just a new chamber with some new vacuum ports added. The heater is capable of heating rotating and linearly translating part to several hundred degrees Celsius. The electrical control and power delivery requirements alone necessitate an additional equipment rack. Incorporating the heater into the design changes many mechanical items so that these parts can accommodate thermal extremes. So, once contract modification was discussed, the design was stopped until the government contract modification was processed. This required Dr. Greer to store this half-developed system in a new location. It also required him to move it to his new production facility.

Section 1. Introduction and Background: Software Approach

Effort Objective Motivation

With increased financial streamlining, the Air Force has desired to extend the lifetimes of aging systems by targeting the improvement of critical mechanical parts. [1] Increasing machine process efficiency as well as reducing process times and scrap rate reduce the overall cost of component part production. A reduction in component part costs ultimately reduces the maintenance of existing air fleet components as well as next generation production of parts. The Air Force Materials Directorate has invested effort in pursuing this endeavor. Techniques to reduce the vibration in machine tools extend from improved work piece fixtures to active feedback of acoustical sensors. A sensor that provides a nondestructive method of detecting machine tool vibration would allow for more optimal feed and speed adjustments to be used with improved part finish and decreased time. Enhancements to process control have evolved that have shown the benefits of control and data collection on many different types of processes; such as InfoScribe process control and data management. (Pat. 5461559) Further development of these capabilities is required so that actual hardware can be economically and efficiently machined.

In designing a coating system, maximum use of off-shelf components produce the highest functionality for a given cost. In order to economically and efficiently coat parts with useful shapes and geometries for direct application testing, an MSPLD machine capable of production quantities is desired. This production quantity machine design implements existing techniques and experimentally verified concepts that have already shown success in preliminary laboratory research. Additional enhancements to basic techniques are rendered in the design so that high reliability production of coated parts is possible.

Technical Objectives Defined

The original technical objectives of the Magnetron Sputtered Pulsed Laser Deposition Scale Up were twofold. First was the development of a Magnetron Sputtered Pulsed Laser Deposition (MSPLD) process capable of producing coated diamond like multilayer heterostructural films over large areas at low process deposition temperatures. These low deposition temperatures are critical for the coating of precision-machined mechanical parts. The objective was primarily to utilize the newly developed Air Force super hard coating technologies in practical applications, such as turbine engine rotor components, precision shafts and ball bearings, rods, and bearing races. This would give the Air Force the capability of practically coating real precision mechanical parts with solid lubricant films of the highest toughness. These films would be of the highest hardness and the most resilient to damage.

The second technical objective was to develop an automated process capable of producing large volumes of coated parts with minimal operator intervention. The production of large volumes of coated parts is necessary to reduce the intrinsic cost of each coating and take advantage of the economies of scale. The MSPLD production capability could be utilized in the case of technology transfer to aircraft component manufacturers, or other cases requiring the production of large volumes of low cost components, such as a war effort. Utilization of such advanced technology would give our soldiers an added advantage in weaponry. Weapon system failures would be less seldom. Component operating margins could be extended into extreme environments. In addition, automation of production could keep costs low by reducing labor while increasing coating consistency.

From these technical objectives, a coating process was devised that would be capable of achieving these objectives in the allotted time frames and costs. The resulting system would be shipped to the Air Force Materials lab as a production ready system. The Materials lab could then utilize this process to demonstrate real technological transfer as well as produce films for volume research and quantity repeatability analysis.

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Unfortunately, the existing system design would need to be drastically changed if it was to accommodate the heater modifications. To finish the system without consideration of the heater would mean that if the heater would be purchased at a later date, the expense would be at least twice what it was if the heater was to be “designed in”. It was not possible to merely “add on” the heater as a separate purchase, but it had to be included in the design of the system. Therefore, in order to save the costs of adding the heater later, development of the MSPLD system was halted until the contract modification was in place.

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Software Objectives

The InfoScribe data acquisition system is the modular system approach that has been demonstrated on many Air Force processes for sensor-actuator connectivity and data archiving and management. Sensor-actuator pairs were identified in the original system design, and hardware-software computer interfaces developed for them. The software utilizes diagnostic programs to evaluate the process as it produces material.

In this case, the InfoScribe software was to now be used as a data server and archive hub for any PC in the lab running the ISX LabVIEW modules. This way, the already purchased hardware could be utilized with a PC provided by the government to control and archive data generated by the modified MSPLD system. The flexibility of the LabVIEW software running the ISX VI modules would provide the additional feature of lab wide data collection. A dedicated network would be required to provide for isolated operation of the ISX VI under LabVIEW. This solution would provide additional security as well, by hardware isolation of the process computers. Examination of the InfoScribe software in data server mode was undertaken.

One of the important things to determine was if the InfoScribe software could perform in a control mode server over a dedicated network. This was done by comparing the software in integral mode to that performance obtained in a remote server mode.

Operation of the InfoScribe software as a data server showed that initial real-time skew figures were in the low tens-of-milliseconds overall for data turnaround and recovery. The InfoScribe data acquisition system was targeted at two additional processes specifically for sensor connectivity. One of these additional processes was a biological sensor trend evaluation. The other process sensoring involves aircraft engine part quality assurance. Companion to these two sensor-oriented processes is an Internet extension module for remote sensoring of the processes via the ISX and LabVIEW software over TCP/IP protocol. The modular nature of the system allowed ready development and inclusion of the TCP/IP Internet support module into the existing system. The InfoScribe data acquisition system is in the process of being upgraded to increase its data bandwidth and provide extended timetag range and resolution. InfoScribe was also upgraded to address operator interface optimizations for process visualization.

InfoScribe data archiving and web based data serving provides WL with a versatile laboratory infrastructure for materials process automation and materials process data archiving and retrieval at the local laboratory process computer and remotely via local area network and Internet connections. InfoScribe software combined with the ISX modules in LabVIEW establishes the capability to custom design a data acquisition system by assembling reusable software components that share process data parameters via network communication methods on a target process control computer.

Software baseline comparative study

The architecture of the baseline integral data acquisition and control system must be investigated before the software can be converted from a single computer system to a network based multiple computer network control system. This section discusses the work done to research this modification. The traditional InfoScribe integral system was used as the baseline comparative architecture to compare software control with a TCP based network control system. The architecture of the baseline integral data acquisition and control system is illustrated in the Figure 1.

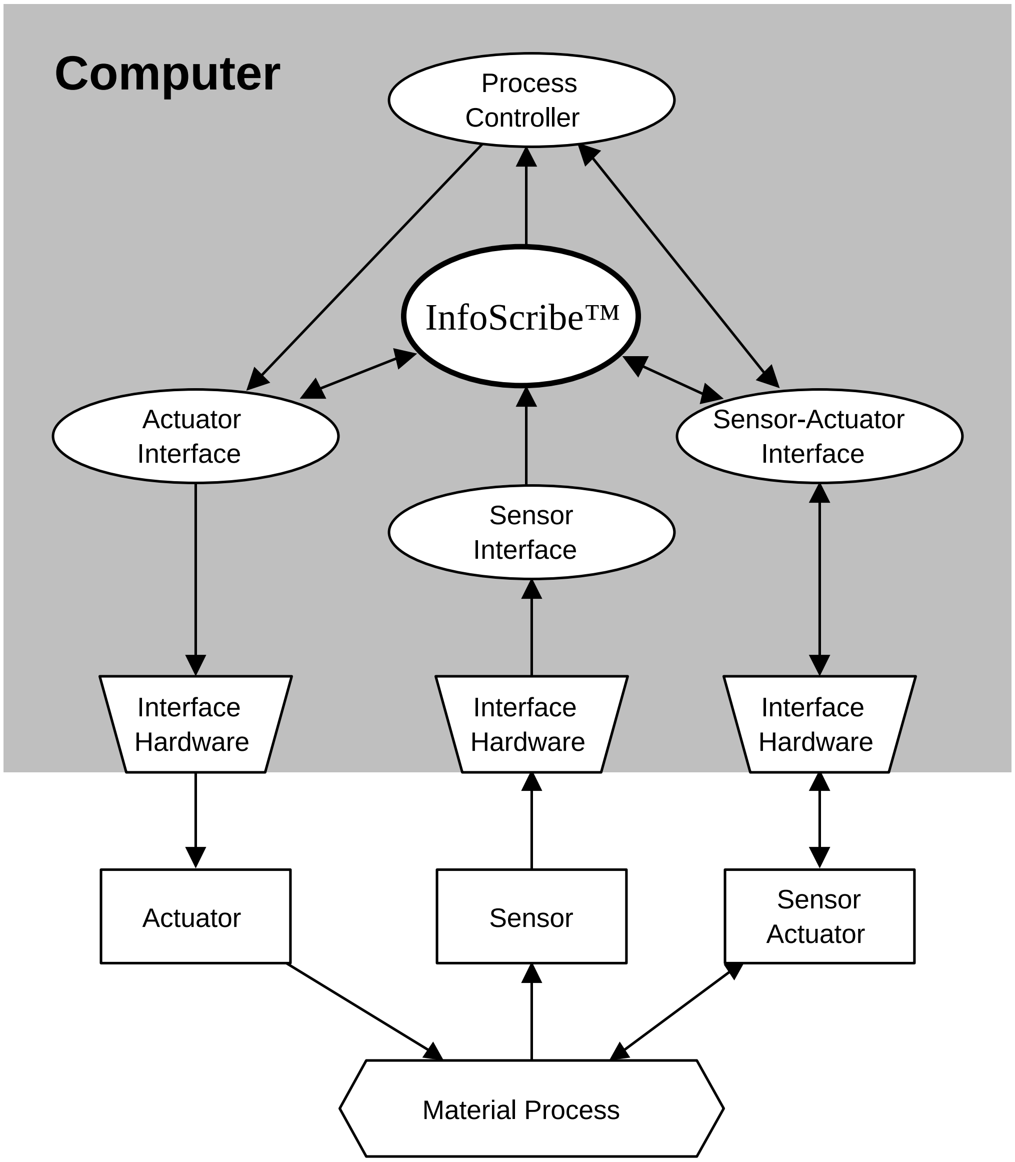

Figure 1 Integral Baseline Hierarchical Structure

The software components in Figure 1 are represented by elliptical graphic elements. The interconnections were tested and first replaced by a microkernel dispatch based architecture, then separated out then separated out with TCP interfaces to evaluate system feasibility of a network-based system.

The trapezoidal elements represent the hardware interface between the sensor and actuators of the materials process and specific software interface support components. This forms the boundary between the physical materials processing equipment and the abstraction layer of hierarchical materials process automation. The central graphic element is the InfoScribe data acquisition module that performs data archiving and graphical time base plotting display of the process data. The topmost graphic element is the materials process controller. This element represents the supervisory control functions needed to effect the progress of the materials process toward the desired materials characteristics of the product. The communication links among these individual software modules is such that additional sensor or actuator interface modules or additional process enhancement modules can be added by simply loading the module into the working directory of the process control computer.

An overview of the integral baseline hierarchical structure in Figure 1 is described next. Each component is annotated to show interaction of the structure.

1. InfoScribe: The core component of a hierarchical data acquisition and control structure that supports data transfer among modules through a local area network based communication protocol to each subscribing module. It provides dual redundant data storage and retrieval associated with internal computer hard drives. This archive supports the *in situ* streaming of process data to non-volatile storage media. Also, supported is the expedient retrieval of any data required by companion modules. It also provides graphical time based display of process and user data. Provides direct user manipulation of data for viewing preferences along with exporting of user selected data or direct linking to commercial analysis software.

2. Sensor Interface: Interface module that supports unique process instrumentation computer input interface and configuration requirements.

3. Actuator Interface: Interface module that supports unique process actuator computer output interface and configuration requirements.

4. Sensor-Actuator Interface: Interface module that supports unique process instrumentation and actuator computer input/output interface and configuration requirements.

5. Process Controller: Execution module to control a given process toward a desired goal. This module combines sensor input data with process prescriptions to determine necessary process actuation to achieve the goals of the process through fixed schedule, closed-loop, or adaptive feedback control.

6. Interface Hardware: Commercial data acquisition hardware composed of plug-in computer interface boards for serial, coaxial, parallel digital and analog input/output control of the required material process instrumentation.

7. Sensor/Sensor-Actuator/Actuator: The required material process instrumentation and control equipment that is attached to the material process.

8. Material Process: The material process of interest.

Baseline Software Flexibility

The flexibility of this system has been demonstrated by the differences in the installations that currently exist throughout the Air Force as well as several other Government research labs and commercial corporations. These installations include different combinations of sensor-actuator interface modules such as Proportional-Integral-Derivative (PID) temperature interface modules, shutter control interface modules, ion gauge pressure interfaces modules, partial pressure interface modules, pump control relay interfaces modules, stepper motor interface modules, and general purpose analog-digital voltage interface modules. Process control modules specific to Molecular Beam Epitaxy (MBE) are in place and general-purpose control modules for Pulsed Laser Deposition (PLD) and Chemical Vapor Deposition (CVD) are in use. Other modules include supervisor control for automatic process control. File handling and generation on a day-by-day basis are also possible using web based client techniques. There are in use special user interfaces to account for operator security and accountability. The interface modules to other software is available now to interface with other custom control systems written in other languages, such as LabView.

Many InfoScribe installations utilize the same software modules for their InfoScribe data acquisition systems. These modules include InfoScribe, PIDcontrol module, ShutterControl module, IonGauge module, GrowthControl module, InfoSupervisor module, and *In situ*Messenger module, as well as other software. The added heater can be controlled by PIDcontrol. Other software, such as LabView, can accommodate material growth recipes from any data file by acquiring an automatically generated list of InfoScribe system control parameters specific to the respective machine. These control parameters are defined for each unique sensor-actuator software interface upon definition and for appropriate process control modules. InfoScribe can operate in a non-control mode to monitor any system to provide process tracking and accountability unavailable with any other vendor's systems.

Additional installations such as PLD and CVD also use InfoScribe with the InfoSupervisor module, PIDcontrol module, *In situ*Messenger, and a module called ISX that is designed to interface with LabView applications. The ISX module supports data communication between the InfoScribe data acquisition system and pre-existing LabView developments. The ISX module is also very useful by providing a link to the LabView development environment for new sensor-actuator development. This system of software was tested on a single computer, and found to have the following transit times for information exchange using apple events and dispersed communication architecture.

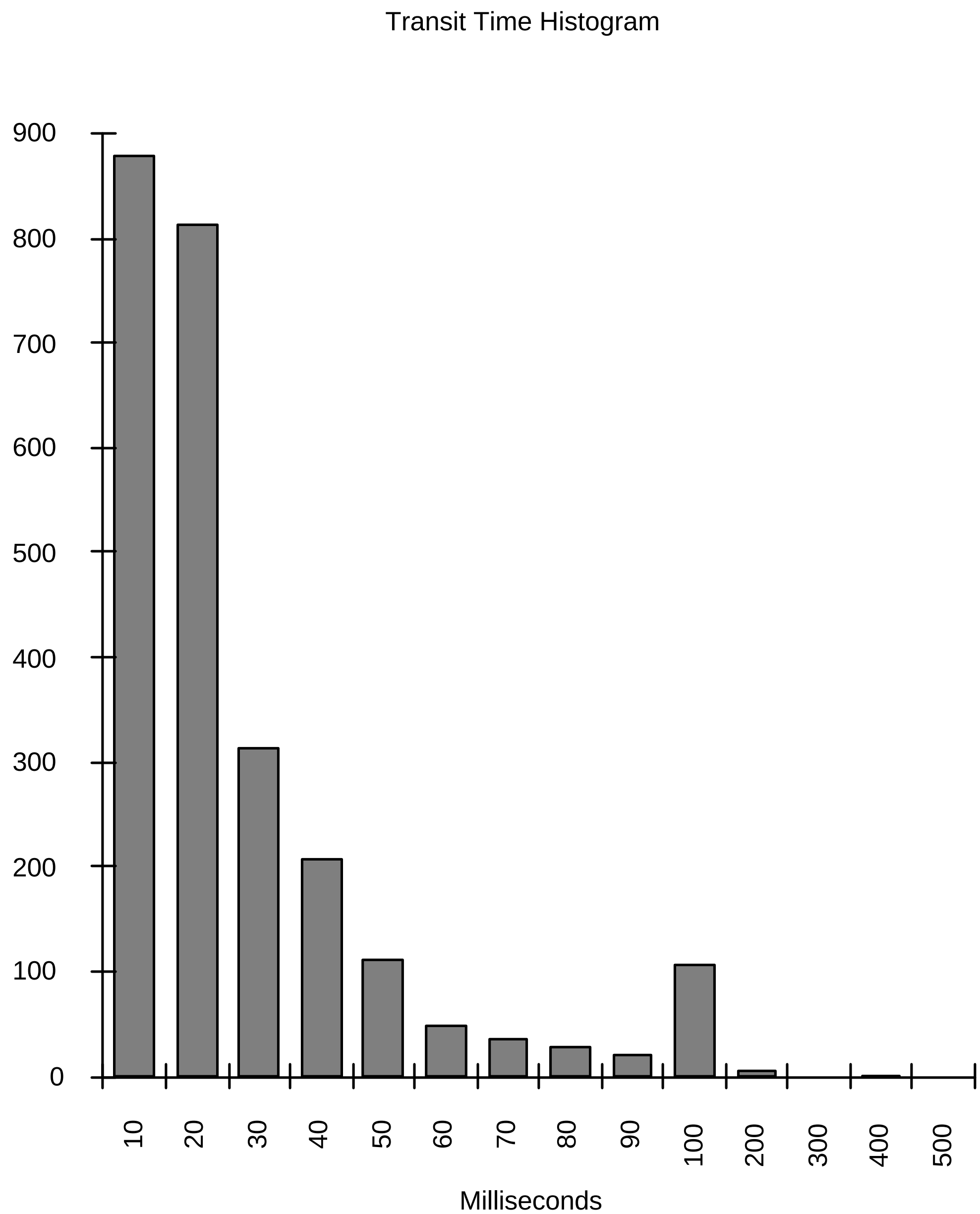

Figure 2 Baseline Software Latencies

As can be seen, the transient time for communication between software modules ranges from 10 to 100 milliseconds. This limits the software usefulness to real time control that is on the second interval.

The statistical mean and variance are shown for Figure 2. As can be seen, communication occurs on the mean of 34 milliseconds and a one-sigma standard deviation of 28 milliseconds.

Mean	34 milliseconds
Standard Deviation	28 milliseconds

Table 1 Network-Based Communication Latency

After a new sensor-actuator computer interface has been developed in the LabView development environment, the graphical LabView development diagrams can be translated into efficient high-speed C/C++ code to remove the large computer system resource burden imposed by LabView and allow distribution of the new module to multiple materials processing computer systems.
Four other InfoScribe installations off-site from AFRL/ML utilize InfoScribe with the InfoSupervisor module, *In situ*Messenger, and a special sensor interface module called TCM3Link for monitoring partial gas pressures of oxygen and carbon dioxide of both human patients and laboratory animals in hyperbaric oxygen therapy. Two of these installations for hyperbaric oxygen therapy are located at Wright Patterson AFB while the other two installations are at Brooks AFB and Travis AFB.

The success of network based inter-application communication (IAC) in the InfoScribe system evolved from the robust object-oriented inter-application communication structure provided at the operating system level of InfoScribe's design platform. The design platform for InfoScribe uses the MacOS and operates on both Motorola 680x0 processors and on IBM/Motorola PowerPC processors. The former processor is a complex instruction set computer (CISC) design and its clock speed is limited to less than 100MHz. The later processor is a reduced instruction set computer (RISC) design and its clock speed is currently available at 225MHz with future projections to 500MHz. The significance of these processors is that they are the core of low-cost desktop and mini-tower personal computers and they provide the inter-application communication latency time shown in Figure 2. The condensed latency time in Table 1 shows a mean message transmission time of 34 milliseconds. With the standard deviation of 28 milliseconds, the worst-case transmission time is only 62 milliseconds. The best-case transmission time is mere 6 milliseconds.

These message transmission times are the basis of the inter-application communication efficiency built into the InfoScribe system. Equally important is that the network based communication architecture is inherently local area network (LAN) capable. This gives the InfoScribe data acquisition and control system the ability to locate a compute intensive operation such as material process modeling on a separate computer for unimpeded processing while data acquisition and process control are handled by the main materials processing computer. This network capability has also been utilized for remote monitoring of a materials process via LAN in the researcher's office separated from the materials processing lab. The network capability also includes Internet access via TCP/IP protocol for long distance monitoring of processes located in physically separated facilities.

The technical objectives of the software developed for the MSPLD system with modification became threefold. First, the mechanics of the InfoScribe data acquisition system were enhanced to exploit the data handling capacity of the latest low-cost RISC processor based computer systems through the implementation of a micro-kernel design for the InfoScribe data acquisition system. Second, the modeling methodologies explored for data acquisition, control, and noise characterization and discussed in the original proposal were refined and implemented as InfoScribe process modeling and controlling modules improved. Third, the benefits of these modeling methodologies as rendered in active modeling and controlling modules were established in the assessment of improved materials characteristics and yield from AFRL/ML materials processes. A parallel assessment of commercial installations also accompanied the commercialization of effort of the InfoScribe data management system. These three points opened up the MSPLD process as a viable production system.

Section 2. Objective and Approach: Software Generic Design

The software development was conducted over a period of two years with several modifications during this time. In order to accomplish the technical objectives described in the previous section, the program was divided into two tasks.

Task 1. Design the data acquisition and process control system around a general-purpose micro-kernel that utilizes minimum platform specific operating system features and implements pre-emptive task scheduling on a priority basis. This software would be used as the data server for the MLBT dedicated network.

Task 2. Assess the process modeling and control system by design, testing and engineering, through the addition and substitution of the modular process modeling and control components of the system on the process. Develop the ISX interface to LabVIEW to provide existing system integration.

Task 1. Design Micro-Kernel Data Acquisition Process Control System.

Three-dimensional process model visualization was explored as an ancillary user interface to real-time data acquisition. Although parameterization rules may be required to support effective three-dimensional process model visualization, the need for flexible InfoScribe data viewing capability was indicated. These multiple viewing capacities included the current time based data plots along with two-dimensional and three-dimensional graphical views as well as textual user notes. The capability to support multiple and varied data viewer formats came from an expansion of InfoScribe's data handling bandwidth, fully automated communication addressing, and pre-emptive task scheduling based on module functionality defined priority level.

These new features were implemented in the development of an InfoScribe micro-kernel. The micro-kernel supports all of the existing features that the present InfoScribe system provides through its network based inter-application communication. The InfoScribe kernel will maintain Internet connectivity through its existing TCPLink module and LAN support will be maintained through a similar companion module.

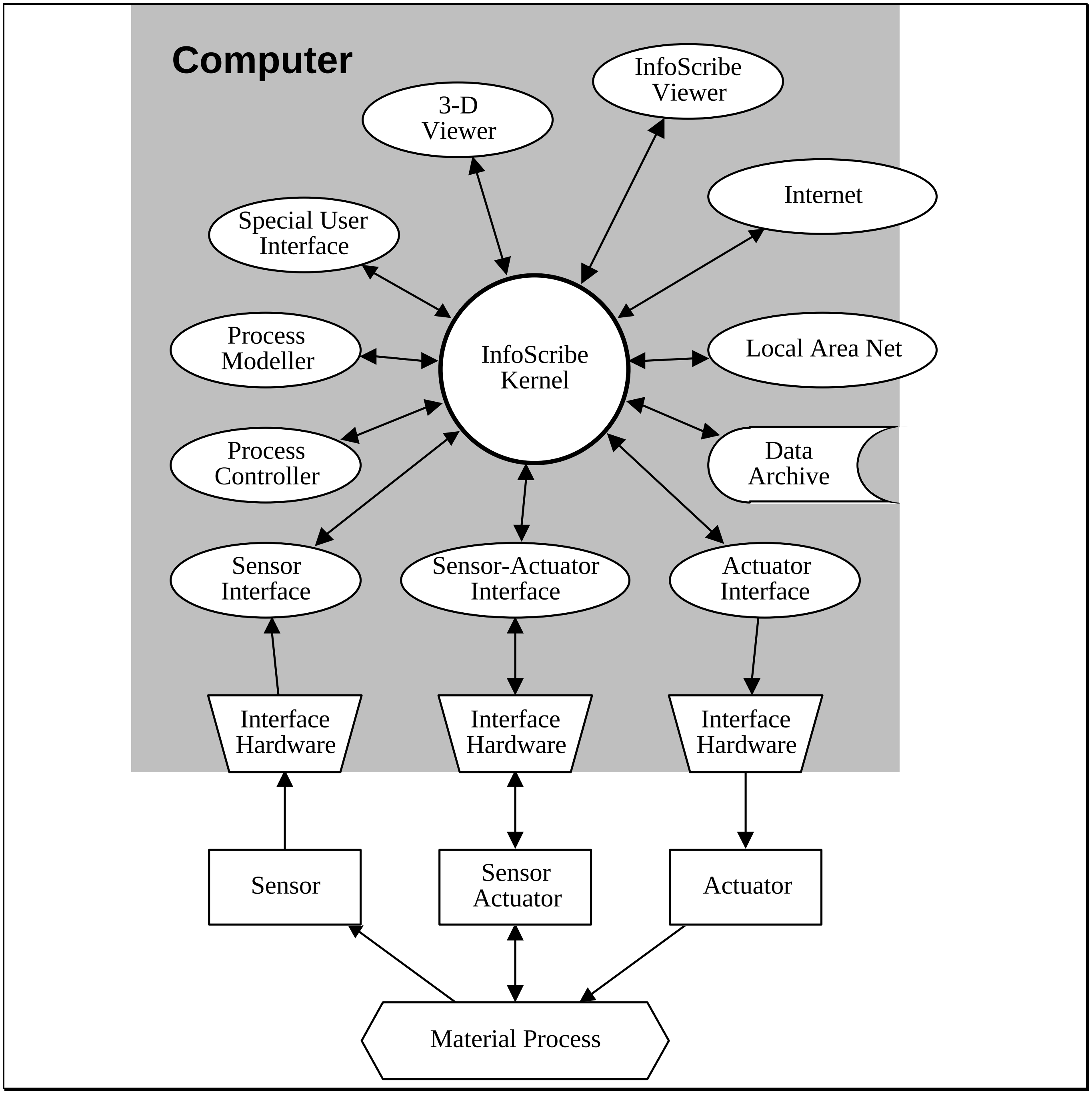

Figure 3 Direct Dispatch Micro-Kernel Structure

Shown next is an overview of Direct Dispatch Micro-Kernel Structure in Figure 3. The software and hardware structure are integral.

1. InfoScribe Kernel: A sub-system kernel that supports data transfer among modules through direct dispatch to each subscribing module and near real-time process scheduling through assigned priority levels.

2. Data Archive: Dual redundant data storage and retrieval associated with internal computer hard drives. This archive supports the *in situ* streaming of real-time process data to non-volatile storage media. Also, supported is the expedient retrieval of any data required by companion modules.

3. InfoScribe Viewer: Graphical time based plotting, 2-D, and textual display of process and user data. Provides direct user manipulation of data for viewing preferences along with exporting of user selected data or direct linking to commercial analysis software.

4. 3-D Viewer: Graphical user interface for viewing data in unique three-dimensional groupings for improved process visualization. Three-dimensional visualization of megabytes of data is possible through variations in shape, size, grouping, and color.

5. Sensor Interface: Interface module that supports unique process instrumentation computer input interface and configuration requirements.

6. Actuator Interface: Interface module that supports unique process actuator computer output interface and configuration requirements.

7. Sensor-Actuator Interface: Interface module that supports unique process instrumentation and actuator computer input/output interface and configuration requirements.

8. Process Controller: Execution module to control a given process toward a desired goal. This module combines sensor input data with process prescriptions to determine necessary process actuation to achieve to goals of the process through fixed schedule, closed-loop, or adaptive feedback control.

9. Process Model: Assistance to the process controller by augmenting adaptive feedback control. The model would be based on mathematical simulation of real process data, neural-networks, or other artificial intelligence methods based on *in situ* and/or *ex situ* data.

10. Local Area Net: Multi-processing network support to off-load computer intensive processing tasks, large bandwidth data acquisition, or user interface isolation from time critical process control.

11. Internet: Wide area network support for remote data file transfer, remote query of InfoScribe data archives, and remote monitoring of widely dispersed processing sites.

12. Special User Interface: Custom interface to support user unique process requirements. Supported by generic access to the InfoScribe Kernel, special interfaces provide customized display and user input control. Within the special user interface, foreign data files from physically isolated sources may be imported into the InfoScribe data file format to combine separate disparate sources and utilize the process data visualization available with InfoScribe.

13. Interface Hardware: Commercial data acquisition hardware composed of plug-in computer interface boards for serial, coaxial, parallel digital and analog input/output control of the required material process instrumentation.

14. Sensor/Sensor-Actuator/Actuator: The required material process instrumentation and control equipment that is attached to the material process.

15. Material Process: The material process of interest.

This system was developed and tested. A direct comparison to the previous system was then done to compare the previous architecture to the kernel design. A ten-fold decrease in latencies was observed. This is shown next.

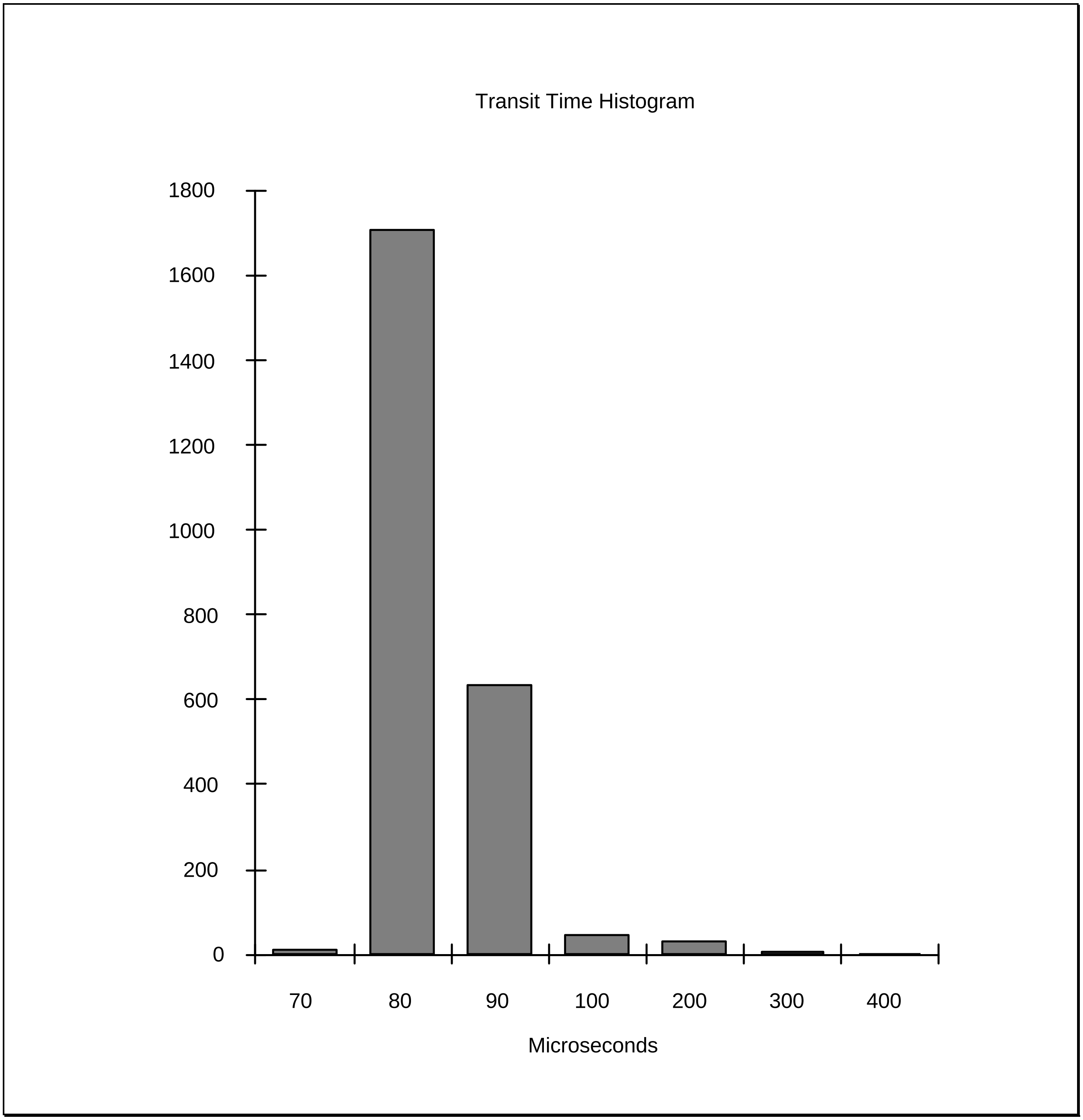

Figure 4 Direct Dispatch Communication Latency

Also shown here is a tabulated form of the statistical mean and variance of 1000 data dispatch communications as shown in Figure 4.

Mean	91 microseconds
Standard Deviation	30 microseconds

Table 2 Direct Dispatch Communication Latency

The remaining communications for modules located directly on the processing computer with the InfoScribe kernel will be supported through direct dispatch messaging. Figure 3 details the structure of the micro-kernel based InfoScribe system. All communication is routed directly through the kernel just as the network based inter-application communication was routed through the computer operating system. With the micro-kernel design, the InfoScribe viewer would not have to be located on the host process control computer, but could be located on a remote computer or even on both or multiple computers located throughout a laboratory facility. By the same token, process modeling could be located on one or more remote computers dedicated to compute intensive modeling activity in a parallel processing paradigm.

Pre-emptive task scheduling will be based on a non-interrupt context switch among the modules linked with the InfoScribe micro-kernel. All of these modules, by design, will make quasi-regular calls to micro-kernel services such as *'GetData'* and *SetData'*. During these calls to the micro-kernel, any module that has installed a procedure to be called periodically will be accessed. The rate that such a procedure is accessed will be determined by a periodicity level requested by individual modules. During regular calls to the micro-kernel by the current active client module, each suspended module's periodicity level is checked for periodic calling eligibility. When a suspended module becomes eligible for periodic calling, the current active module is pre-empted so that the periodic procedure of the suspended module can be called at a non-interrupt state and allow that module to have full access to all computer operating system resources. This pre-emptive task scheduling will provide regular processor time to all modules that need to service computer hardware interfaces for real-time data acquisition and process control. This pre-emptive task scheduling will also support data displays that need to be updated at regular rates that are much slower than the actual flow of new data thereby minimizing excessive computer display redrawing.

Data server architecture

The direct dispatch communication latency is represented in Figure 4 to show the improved message transmission time available by using the micro-kernel design. Whereas Figure 3 and Table 1 for the network based communication has a mean latency of 34 milliseconds, Figure 4 and Table 2 show that direct dispatch communication has a mean latency of only 91 microseconds. Also, since the micro-kernel communication is isolated from operating system dependencies, the direct dispatch communication time will decrease directly as computer processor speeds increase to allow continual system performance enhancement as computer hardware is upgraded. The direct dispatch communication bridge to network communication will be handled through a module specific to TCP/IP Internet protocol and another specific to LAN protocol. While the TCPLink module already exists to support the Internet protocol, a new module will be developed from the network based communication system to support LAN protocol. Figure 5 represents a micro-kernel based InfoScribe system supporting both LAN and Internet connectivity to remote computers. Computer 1 is the host computer connected directly to the materials process. It utilizes interface hardware to connect to the materials process, it supports the data archive, and it supports a typical collection of InfoScribe data acquisition and process control modules.

Computer 2 is a remote computer located on a LAN associated with the computer 1. Computer 2 is utilized as a remote monitoring station that is either in the researcher's office or is located outside of a clean room that might contain computer 1. Computer 3 is on the same LAN with computer 1 and computer 2, but instead of a data viewer, it is occupied with a compute intensive process-modeling task that is used to support the process controller on computer 1 in a parallel processing paradigm. Computer 3 also has local access to a materials processing database to aid in the modeling task. The location of computer 4 could be in the same building as the first three computers or it could be across town or across the country. Computer 4 maintains data viewers for a researcher's use to study the progress of a remote materials process and maintains a process model to test local materials processing concepts.

The design of the InfoScribe micro-kernel is the critical component of data acquisition and control system. The foundation for the micro-kernel will be based on the current network based communication standards and the file handling architecture within InfoScribe. Through the evolution of InfoScribe and the goal to keep development schedules on time, the application-programming interface (API) for the InfoScribe micro-kernel will be kept very simple. Basic access to the micro-kernel will require the software module developer to implement only the six-procedure calls listed below:

1. Initialize()
2. InstallPeriodicHandler(PeriodicHandler,periodic level)
3. GetList(header specifications , tag)
4. GetData(header specification, data)
5. SetData(header specification, data)
6. Terminate()

The first procedure *'Initialize'* is called when the module is started to access the micro-kernel. If the kernel is not running, it will be started. If the micro-kernel cannot be found, then the module will function in a standalone mode if applicable. The second procedure *'InstallPeriodicHandler'* will install a periodic time handler to enable the module to be called by the micro-kernel as a pre-emptively scheduled task. The third procedure *'GetList'* is used by the module to access a list of all control parameters available within the context of an installed InfoScribe data acquisition system. The fourth procedure *'GetData'* will be used by the module to access a specific control parameter's data as referenced by its header specification. The fifth procedure *'SetData'* will be used by the module to assign data to a specific control parameter as referenced by its header specification. This assigned data will be automatically routed to the module that owns the particular control parameter. The sixth procedure *'Terminate'* is called when the module is stopped so that the connection to the micro-kernel terminated efficiently. It is possible for a software module to encounter an error condition where it will stop unexpectedly. Because this is a possibility, the micro-kernel will maintain error checking functions to ensure that fault conditions are handled robustly. Two additional procedures available for a module to schedule automatic reception of process data are:

7. InstallListHandler(ListHandler)
8. InstallReceiveDataHandler(ReceiveDataHandler)

The last two procedure calls will enable a module a limited amount of priority for batch handling the procedures GetData and SetData where several control parameters may need to be handled as a group. These procedures are:

9. SetHighPriority()
10. SetNormalPriority()

The procedure *'SetHighPriority'* will temporarily disable micro-kernel calls to pre-emptive task scheduling. The effects of this call will time out to prevent permanent disabling of task scheduling. The procedure *'SetNormalPriority'* is used to reset the effects of *'SetHighPriority'* and is meant to be called immediately after high priority is no longer needed.

These details described for the InfoScribe micro-kernel are the result of performance testing and actual use of the existing InfoScribe data acquisition system. The designs were based on reducing the cumulative data propagation delays through the data acquisition system as the data is delivered to the computer hard drive for archiving. The micro-kernel design will provide more than two orders of magnitude increase in data transmission speed such that the slowest component of the computer data acquisition system remains at the computer hard drive.

The simplified system level program interface for accessing the micro-kernel as opposed to the current network oriented program interface will reduce development time for new sensor, actuator, modeling, and control modules. The simplified program interface will also speed the delivery of a software development kit to commercial and government users who have a need for confidential or proprietary module development. The multiple computer architecture is shown next.

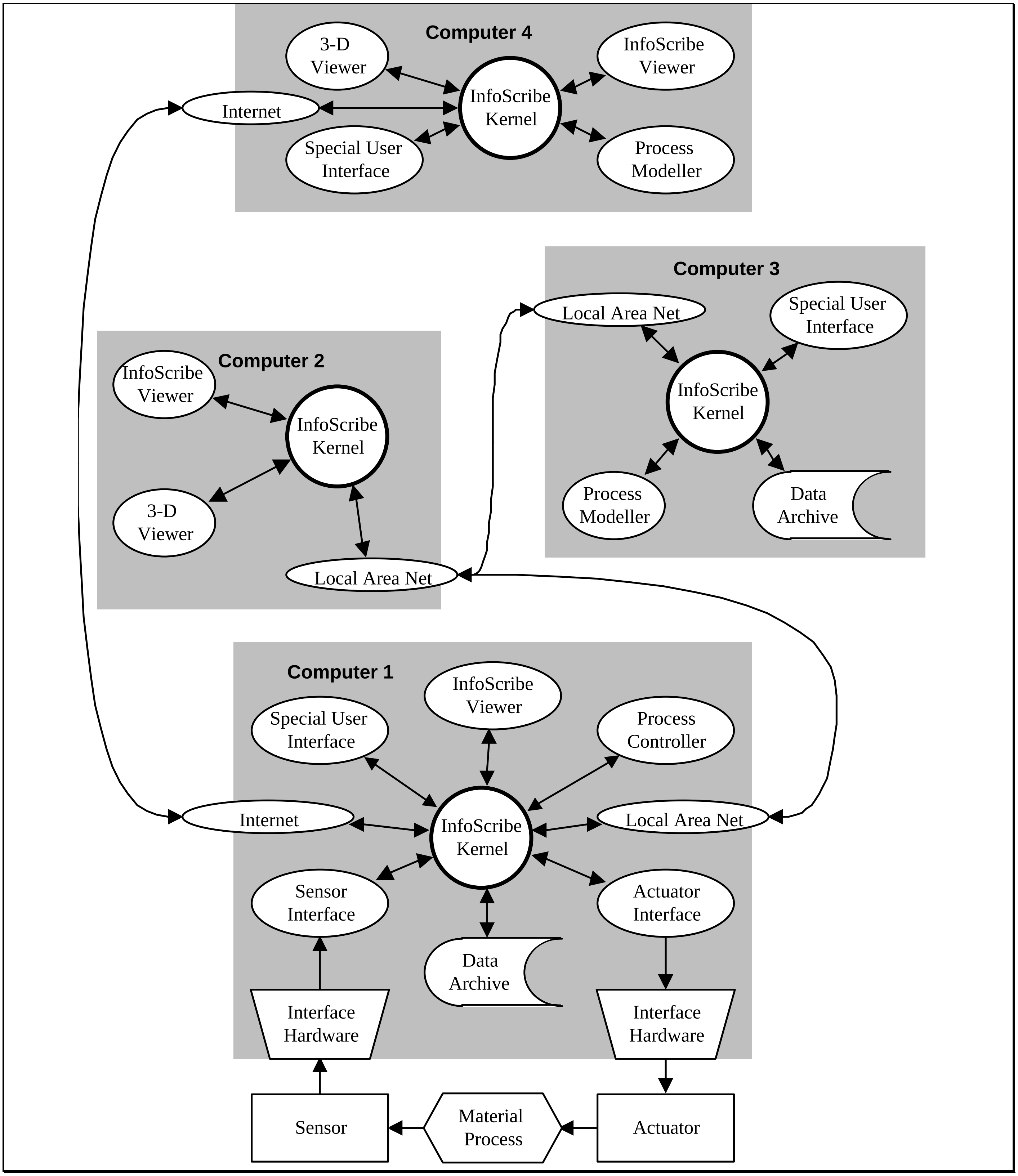

Figure 5 Local Area Net and Internet Connectivity

As can be seen, several processor tasks can be separated out across the network, providing distributed architecture advantages. A process control activity can be taking place in computer one, while several other tasks, such as real time modeling, three-dimensional viewing, statistical analysis, and other numerous activities can be occurring across the network. Only when there is a change in the data will it be transferred over the network.

Task 2. Assess the Process Modeling and Control System

The processes in the MLBT complex must contain a full complement of necessary instruments so that automation via computer, combined with data acquisition and control can commence. It is from this complete sensor and actuator suite that a virtual materials processing environment can exist. Processes that do not incorporate sensors and actuators must also be modified to accommodate the sensing and control of process parameters necessary to accomplish the data archiving information needed to totally exploit each process run. Furthermore, while additional instrumentation and process functionality is always desirous, the design and engineering solutions are different for each process. A unique solution is required for each process. It also becomes important to carefully instrument each process, so that the information collected will be accurate, and, thus, can be disseminated and utilized with little error.

Undertaking the task of automated information dissemination and process control will require the conceptualization, design, engineering, testing and debugging of complex integrated hardware and software solutions. In this case, a most economical approach is through modularity. The need for modular solutions allows for multiple process reusability, such as a specific temperature control software module that can control temperatures from MOCVD reactors just as well as MBE Knudsen cell temperatures. A suite of modules needs to be created that will permit accomplishment of this task with a minimum of effort duplication. Each process instrumentation suite can then be approached by the addition and substitution of modular software and hardware components. The modules required must accommodate a diverse functionality, ultimately limited by the particular instrument restrictions that are imposed by the instrument hardware.

In a further attempt at economical utilization, the use of existing process instrumentation as well as existing LabVIEW process control code was pursued wherever possible to minimize cost expenditures on additional equipment, although this cost reduction will only be attempted without sacrifice of functionality. Currently, the processes in MLBT support the instrumentation, control, and automation of several in-house process instrumentation. The instrumentation of materials target efforts will possibly require each process to have additional software development and hardware purchases to support the full incorporation of that particular process to the network.

Process Instrument Automation Design

Automation of a process to achieve virtual material research requires that process parameters necessary to generate the desired material be known. This is seldom the case. There is usually a discrepancy as to what process parameters are important in altering the produced material and which are not. In addition, if parameters are known to be significant, the parameter control ranges are seldom fully explored. Usually a fixed group of settings is all that is used to make a material. Instruments that can sense these disputed parameters thus require that a suite of sensors and actuators be specified that are capable of a wide range. It also is imperative that in specifying the sensors and actuators, full expert knowledge of how the process is to function is made available. A working synergy is necessary between the process experts and the specifications engineer. It is only then that a group of sensors and actuators can be determined that will fully utilize the physical principle of the process. This task is depicted in Figure 6.

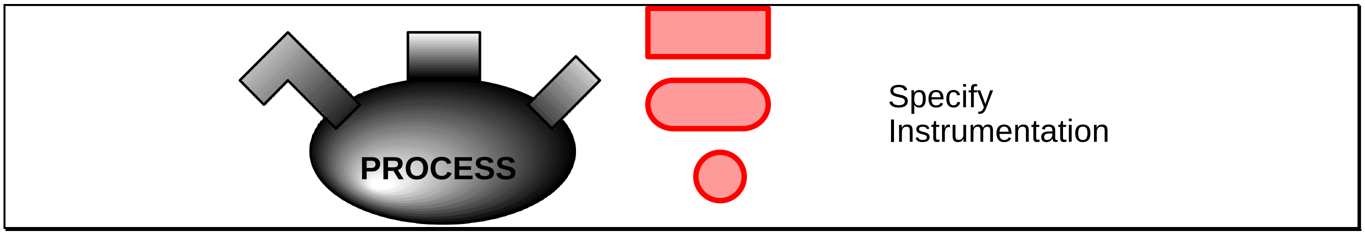

Figure 6 Instrumentation Specification

Once a suite of desired process parameters and subsequent methods to sense them are chosen, integration into the process is needed. This usually requires some physical modification of the process apparatus, requiring machinist skills along with engineering issue solutions. As a rule, it is desirable to modify the process without disturbing operation, so that *in situ* sensing is available. In this way, process parameter measurement can occur without sacrificing the production of material. A graphic depicting this is shown in Figure 7.

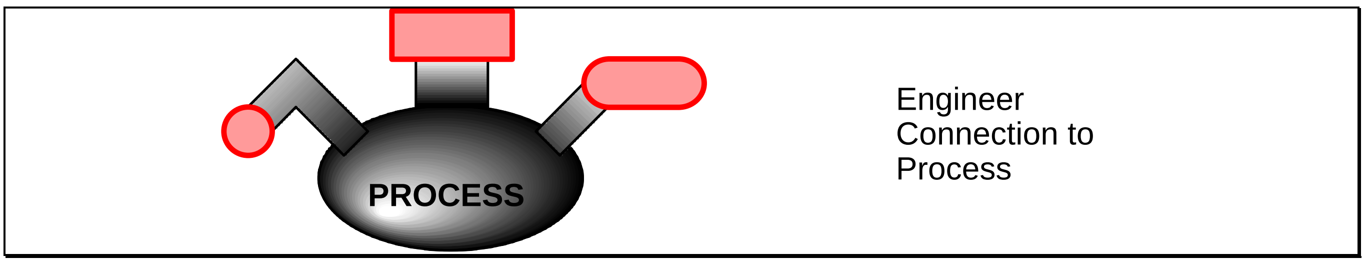

Figure 7 In-situ Sensor Process Integration

Upon the integration of sensors and actuators, it is then necessary to incorporate a computer to create the virtual material process space. Code modules are used for the chosen sensors to allow data collection and process control. This integration will be specific for each process, but utilizes several modules that are combined to achieve the specific functionality of the particular process. The process can be operated under control and the dynamical ranges can be explored. This is depicted in Figure 8.

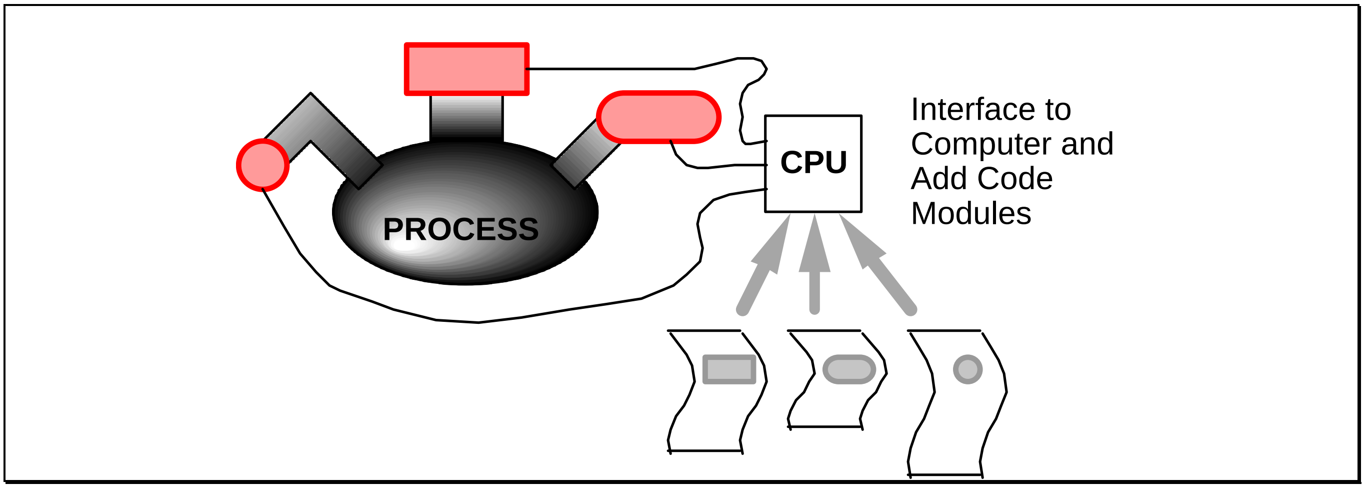

Figure 8 Code Module Installation and Integration

Once the process is engineered for functionality, a group of materials are generated and analyzed. Tests will be performed that pertain to the materials characteristics of interest, and are used to evaluate the functionality of modeling and control software modules. Material will be generated while the process is under instrumentation and control. The *ex situ* analysis of the produced materials will then be compared to *in situ* data to verify the validity of modeling and control modules. The models will then be modified and altered so that they correspond to the process more exactly. This is depicted in Figure 9.

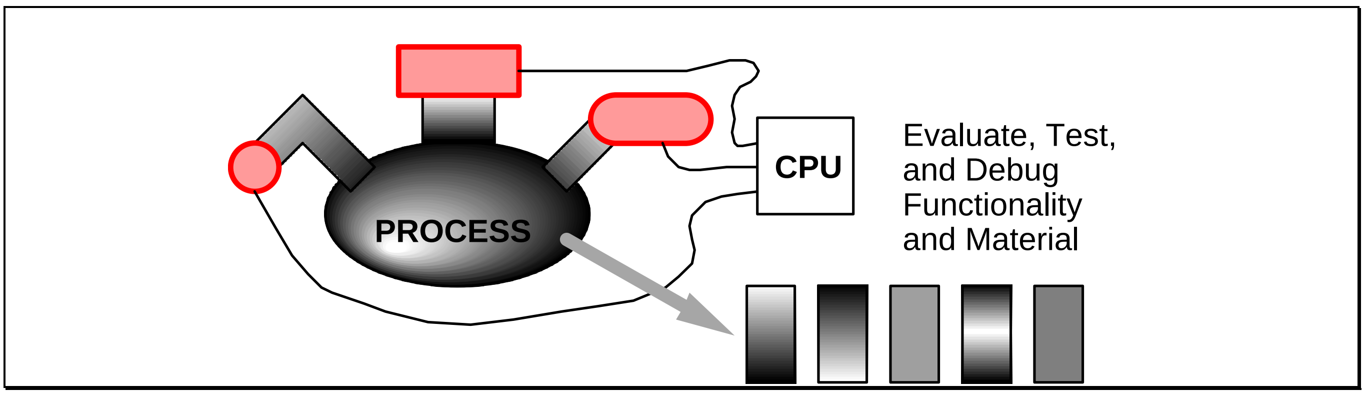

Figure 9 Virtual Process Integrity Verification

The collection of both *ex situ* and *in situ* data from several process runs can then be used to build better understanding of the process operation as well as provide the foundation for virtual materials research. The research can then commence within the computer or over networks without requiring operation of the process.

To implement and further utilize the InfoScribe data server requires that as many processes are added to the server as possible. A list of candidate processes within MLBT that could benefit from utilizing the InfoScribe data server as their data archive follow:

AFRL/MLBT Tasks
MSPLD PLD and DC magnetron hybrid deposition systems: Hardware/software (HW/SW) design, implementation, and re-build for multilayer magnetron/PLD recipe control, including incorporation of magnetrons and mass flow instruments.

Time of flight PLD analysis - HW/SW interface for TOF mass spectrometer to explore plume phenomena, requiring the specification and design of interface hardware and data storage.

High speed tribotester control - HW/SW improvements and functional enhancements as well as code debug/maintenance for experimental data result in storage and retrieval for all tribotesters.

Vacuum Tribotester control - Vacuum Tribotester HW/SW design, build, and code for experimental data result storage and retrieval.

PLD and E-beam hybrid deposition - Ion beam HW/SW, add/rewrite existing PLD recipe control code to accommodate and fully utilize process changes, requiring a complete process redesign.

These processes can benefit from virtual materials research. This will become possible only if the InfoScribe data server serves as a generic common point of data. The success of each process will depend greatly upon the amount of cooperation offered by the indicated deposition process persons. The capability to create a synergistic relationship will ultimately determine the amount of success this data server will have upon the workflow on the group.

Section 3. MSPLD Description of Effort

Before the MSPLD system was designed, some description of the effort is needed. This is to better understand the nature of the process as it was finally constructed and designed. The main driving motivation behind the MSPLD system was the requirements for low temperature films to be developed to meet Air Force needs. Near and long term requirements for tribological thin-films are extremely hard (80 GPa) and very low friction ($M_S < 0.05$) coatings for high temperature (>500°C) environments, vacuum ($<10^{-6}$ Torr) space environments, and low maintenance (10^7 cycles) aircraft propulsion and space mechanism (solid lubricant) applications. From these driving requirements, objective research and focus was determined to fulfill these needs. These are listed next.

PLD Research Objectives

- Investigate *in-situ* sensing methods that determine density and molecular structure
- Develop ‘intelligent’ process design and control methods based upon *in situ* spectroscopy and neurocomputing to enable novel thin film materials

PLD Research Focus

- design and implement process discovery and automation system for DLC/TiC/Ti multilayer tribological thin film pulsed laser deposition (PLD) process
- combine multiple processes, such as PLD and magnetron sputtering deposition, to produce novel multilayer thin-films
- transfer Ti/TiN/DLC multilayer tribological coatings technology using combined PLD and magnetron sputtering methods.

The construction of prototype systems was undertaken, along with much analysis of the resultant film characteristics. These developments are contained in several journal papers, patents, and conference proceedings. An image of a prototype MSPLD system is shown next.

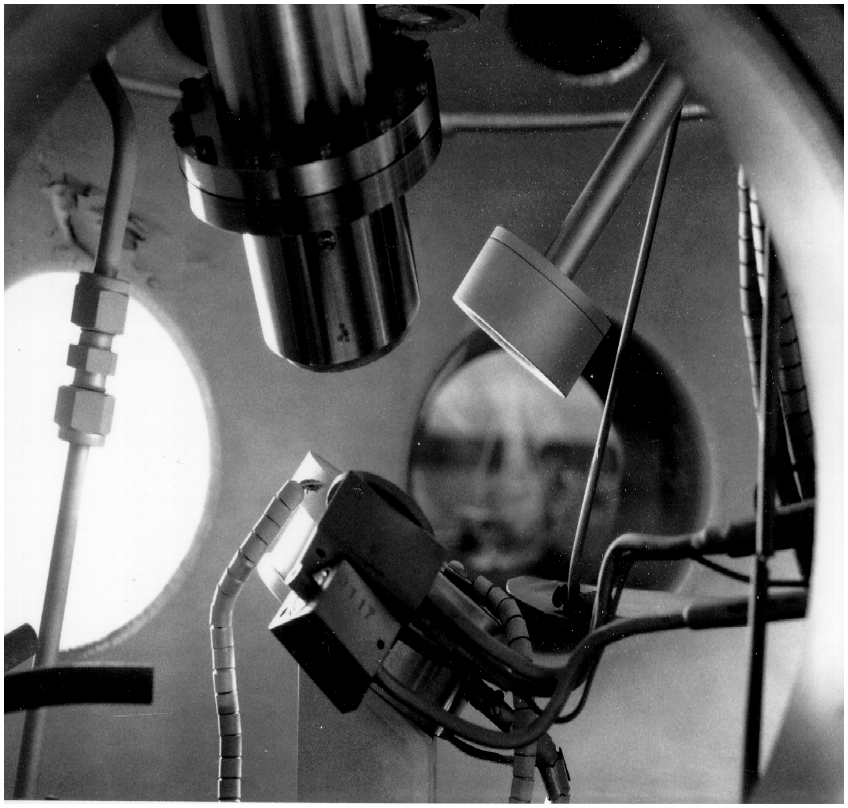

Figure 10 Hybrid Magnetron Sputtering MSPLD - Pulsed Laser Deposition Prototype System

Systems such as this were capable of producing films that met the near and long term requirements for tribological thin-films (or coatings) which are extremely hard - 80 GPa, with very low friction - µs less than 0.05, in high temperature - greater than 500°C, and/or low vacuum - less than 10^{-6} Torr environments (e.g., space), and low maintenance (10^{7} cycles under normal load) aerospace propulsion and momentum control applications. These systems could only produce coatings over small test samples in areas that were less than a square inch. Although this was adequate for research purposes, no actual films were created that were used in actual applications. The performance in real needs was not assessed.

Process Control Sensor and Data Approach

While development of the process was commencing, the development of process control also coincided. The automation of the process became a necessity as multiple layer depositions could only be produced with computer control. Thus, the development of sensor and data process control became important. A firm definition of the process and what the control actually was required to do was undertaken, instead of merely adding on functions as their need was found. A more general view of the process was done.

The past three decades will probably go down in history as the "information age". This is partially due to the development of information computation and storage by the electronic digital computer. Handling and sorting great quantities of information is now relatively easy to do. What previously would have required extensive resources and human endeavor a century ago, can now be achieved on a 500 watt desktop computer and managed by a single human. It is now possible to maintain data on every citizen or household in entire cities, countries, and even the world. Additionally, it is possible to sort and recall this digital data and use the information that is quelled from the data to make large scale decisions on widely varying topics, from tax collection and insurance to ecological and environmental impacts, to marketing and commerce, as well as community and planning. The representation of information has also become pervasive in the field of science and engineering, and has accelerated the generation of new concepts and ideas about the nature of the material existence we endeavor to live in.

Due to this recent advance, our society is critically dependent on the computer representation of time and material quantities, as well as social attitudes and trends. During the “information age”, these concepts have been represented in digital data sets of numbers of finite span and resolution. Consequentially, the conversion of physical or emotional attributes to digital representation necessarily has evoked the principle of approximation.

With the manifold increase of information now being available to a wide audience of scientists and engineers, it is inevitable that misinterpretations and misrepresentations have become more commonplace, with an excellent example of the discovery of “cold fusion” being traced to instrumentation error and drift that was not understood by the scientists making the discovery. Thus, with the increase in information it becomes just as important to convey the accuracy of representation of a given numerical digital representation of some physical phenomena. As well as perceived importance of the numerical representation.

Such is the case in process control, where extended sensors have been installed on processes to yield previously unknown correlation between sensed parameters. This opens up new theoretical enlightenment, such as Laser induced fluorescence in PLD showing increased hardness being related to laser fluence as well as laser wavelength, possibly indicating the domination of photo chromatic effects in laser ablation becoming dominant at shorter laser wavelengths.

Subjective declaration based on representation

As with any codifying of a given physical concept, the declaration of numbers as symbols or measurements involves subjective judgment. In the case of symbols, the numerical representation appears purely arbitrary, as in the numbers on a jersey of a football player, or the numbers that determine the ASCII character table. In the case of numerical measurements, numerical values relate to some physical measure, such as length in feet, or time in picoseconds, with time and length being the abstract concepts, and feet and picoseconds being the numerical manifestation. It has been common practice to define the codification of time with the vibration of a cesium atom, or the length of a foot as being defined by a certain ruling monarch’s physical appendage dimension, but these numerical codification are arbitrary, nonetheless.

In both cases, the information representation is converted, stored, and manipulated in binary form in a digital computer. The computer’s internal hardware architecture ultimately limit the span and resolution of these digital representations, as well as the method of conversion and the effects of manipulation by round off and truncation. These limits introduce error and distort the numbers in a predictable way, and are the study of numerical analysts. What is not discernible by the numerical analysis is the conceptual distortion that is inherent in the subjective judgments used to represent physical matter with numbers.

This subjective judgment affects science that relies on these numbers to verify experiments, to define the occurrence of phenomena, and to experimentally prove a mathematical model theory. A closer look at the scientific method will clarify this further.

The basis of science is the scientific method. The scientific method is widely used by design engineers, physicists, and chemists, applied mathematicians to name a few. The scientific method is described by McGraw-Hill as consisting of six basic human interactions with the world:

“Classification - To accompany conciseness in description, some method of systematizing or classifying the material (or process) is usually adopted.”

In the case of information representation in the computer, the numbers that represent a physical manifestation must be classified into sets or groups that correspond to some arbitrary mathematical model of physical reality or abstract concepts. In the case of physical reality, this is accomplished by grouping sensor data or specific material theoretical parameters together in vectors or sets, occasionally with additional implicit information, such as time implied with a sequence of sensor data. This information is classified in the computer merely by its representation in numerical form.

“Repetition - One of the most potent methods of checking for correctness or truth is repetition.”

In the case of computer representation, the represented numbers must be regenerated for a given physical measurement sensor, or be a reproducible abstraction, such as a fractal. Even though the individual fractal patterns are not reproducible, the abstract concept of a nonlinear dynamic is reproducible.

“Consensus - Another method of checking or confirming the correctness of an observation or report is agreement between different observers.”

Furthermore, the numerical representation of a given physical event must be regenerated by many parties, such as laser induced fluorescence optical emission spectroscopy data being generated of PLD carbon by different peoples of a different culture and a completely different apparatus. An abstract concept’s numerical representation must also be reproducible by any person or algorithm. Otherwise, the possibility of an error or oddity renders the result as a random phenomena, or a particular trait of the computational representation or computer.

“Experiment - One of the most potent tools of many of the sciences, both for the discovery, and for more adequate understanding of existing facts, is the experiment.”

In the case of numerical representation of process parameters, the relationships between numbers can signify a test or series of events that corresponds to a physical event or series of actions.

“Cause and effect - in modern scientific activity, the theoretical analysis preceding the isolation of factors to be experimentally varied is, in the vast majority of cases, predicted on the operation of the law of cause and effect.”

The interrelationships between number groups signify the actions and subsequent reactions that occur in a physical manifestation that exists in a causal universe. The numerical representations have such fidelity that the actual physical event actions and reactions manifest in the numerical representation as interrelationships or mapping between the number groups.

“Measurement - Fundamentally, measurement amounts to description by the use of numbers, but not every use of numbers for the purpose of description is measurement.”

The conversion of physical entities and their dynamical behavior as representations of numbers becomes the key to representation. When the scientific method utilizes computational hardware, the effects of subjective declaration become apparent. One would never train a neural network on the numbers of a football team’s jerseys alone, because training the neural network on the jersey numbers by themselves must have some physical meaning of a causal event in the universe. A subjective meaning such as team jersey numbers and each player’s total yards of ball carrying makes intuitive sense to us, the programmer, but the computer has no concept of this. It makes sense to the programmers of a neural net, to map each player with the number of yards they carry the ball, which is a measurement. Apparently, the mapping must involve an abstract or real measurement. We can even suppose that the running backs, and wide receivers will have the strongest correlation to yards of ball carry, since most plays involve these players carrying the ball the longest distance. Still, the computer algorithm has no concept of this. For instance, each team would have a different set of jersey numbers, and would have a totally different neural net result that would define each team, yet there would be no cognizance of this in the computer unless the program injected their own opinion during the coding process. This lack of apriori information in the computer algorithm limits the development of a very generic algorithm.

One way of infusing a computer’s algorithm with this missing apriori information is by adding numerical representation of the missing information. In the case of processes, this can sometimes be accomplished by adding process sensors. Computational algorithms can then discern if numerical measurements describe cause and effect. The computer is programmed to search out measurements that represent a causal physical universe through interpretation of measurement cause and effect.

Decisions based on subjective declaration

The declaration of what the numerics actually represent limits the computational algorithm’s ability to determine cause and effect. A computational algorithm determines what is desired in the data, and what is not. Let us arbitrarily call the numerics that are desired “signal”, and those numerics that are to be ignored by the computer, or processed out, called “noise”. Unfortunately, this design step of computer hardware, algorithms, and numerical representations manifest the preferences of their sensor and actuator designers, engineers, and software programmers. Remember that the information stored in the computer is a purely subjective declaration. This subjective declaration information loss is best illustrated by example.

Suppose a data set is generated by the simple linear sum of three sampled data orthogonal vectors. These three vectors are a sampled sinusoid, s (m), a sampled “noise” vector, n (m), and a sampled “pulse”, p (m). The resulting data set is then given to three individuals, such that the information is to be stored and interpreted inside a computer, and approximated or interpolated as shown in Figure 10.

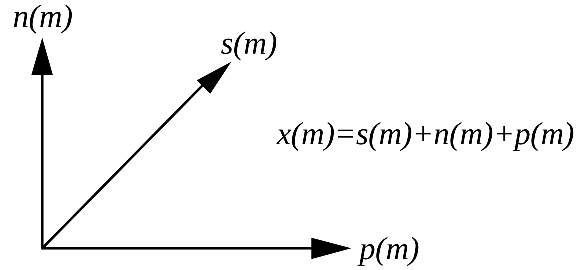

Figure 11 Linear orthogonal sum of three sampled vectors.

Let us suppose that such a data set will be treated by an electrical engineer with a background in power engineering and instrumentation, a particle physicist who has studied impulse phenomena for several years, and a mathematical statistician with a heavy background in stochastic theory. Quite like what is done in a computer, each scientist has no a-priori information as to what the data set actually represents, but is only told to "represent" the "important" information present in x(m). The three independent waveforms are shown in Figure 11, as well as their simple linear sum, that forms the data set given to the three scientists. They must reduce the data set by picking the key attributes of the information based on their experience.

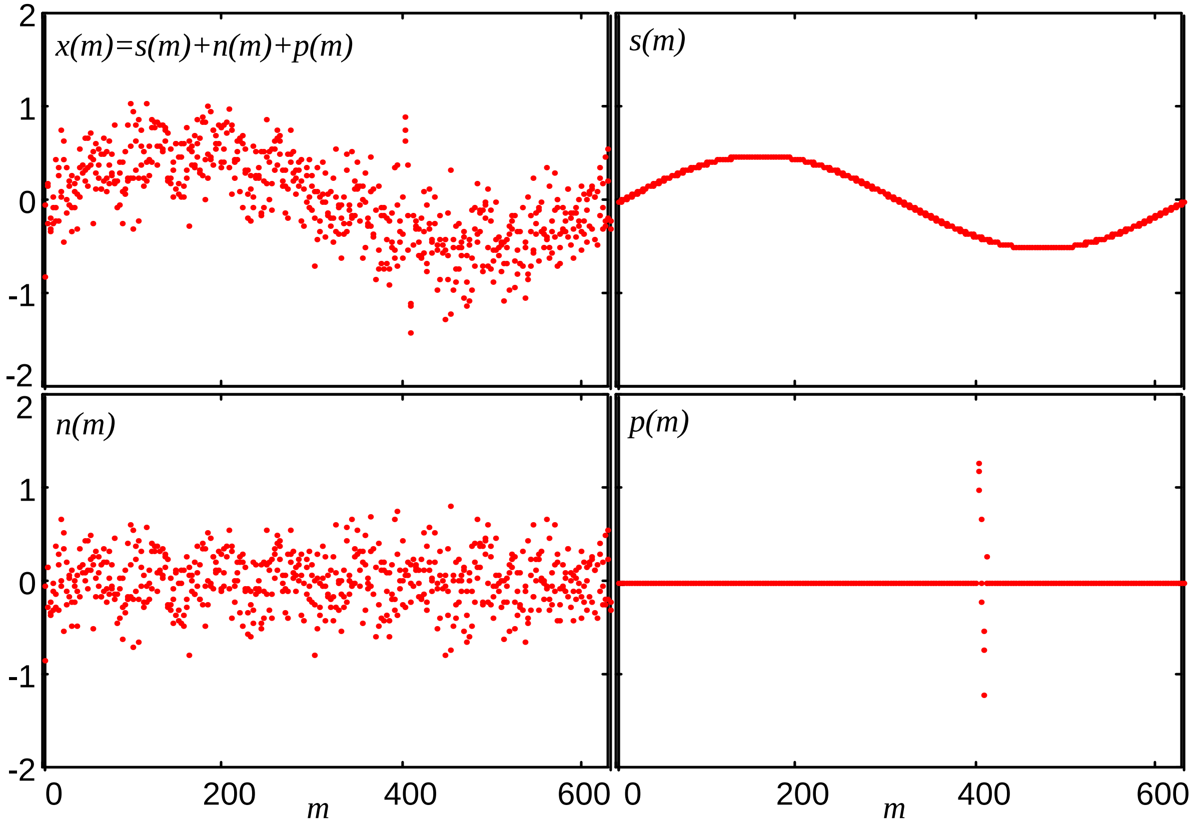

Figure 12 Three sampled data sets and their linear orthogonal sum.

All three will plot the linear orthogonal data set and look for what they consider to be salient features, based on their experience with data. Orthogonalization of the data set by a numerical method could provide each data vector, that is, a sine, a pulse, and a noise component, but that still does not solve the question as to which vector is the important one that contains the true process information, and which are the “noise” components that can be discarded. Below is a synopsis of their rationale of developing an “important” computer data set:

The electrical engineer - This particular hypothetical electrical engineer immediately notices a periodic component with a noise component. They immediately disregard the transient and the “noise” as perhaps a switching spike, and signal noise that is picked up on the instrumentation, or sensor-generated. They focus on the sinusoidal pattern as significant, and determine that is the key attribute of information that represents a physical manifestation of interest. This is possibly due to the extensive periodic waveform schooling that most electrical engineers undertake. Based on estimated dynamics, they develop a sophisticated dynamical filtering algorithm, and end up with a waveform that is a close representation to s(m).

The particle physicist - This hypothetical particle physicist immediately notices the “spike” that occurs around m=400. They disregard the periodic component as perhaps an alternating plasma generation artifact or coherent detection interference, as well as the “noise” component as some kind of insignificant electron noise. They “deconvolve” the data set, and focus on getting the transient so that it’s width and height can be measured. After extensive manipulation of the data, they end up with a representation that is close to p(m).

The mathematical statistician - This particular hypothetical mathematical statistician focuses on the distribution as well as other statistical moments of the data, and decomposes the numerical set into linear orthogonal components, with statistical moments of each orthogonal vector. They may also find that there is several periodic components in the pseudorandom data set, n(m), and consider all these data attributes to be significant.

The digital computer has stored and manipulated the information in all cases, but since no apriori information is available, it is quite likely that different representations will manifest themselves when done by different people. The scientists have no apriori information about the data set, so they “impose” their own experience when they manipulate the data, and end up with three different data set information opinions.

A more prudent scientist might only present the data as a table, and since there is no information about what the data represents, they do not induce their own opinions, but merely save the data until more information can be gathered as to what the data set actually is supposed to represent. Then they can impose their subjective manipulations based on this apriori information. The numerical representations in this case are given as the best technically possible, and stored in time as they occurred. There is no subjective distortion of the numerical representations that would further disrupt the information provided by the sensors. There are also no subjective assumptions about the significance of the data, either. The information is merely stored as it is received.

The point of this example shows that whenever numbers are manipulated in algorithms, it is possible to inject many various methods of interpreting them. This is the problem with information approximation. There is always the glaring possibility of throwing away the wrong stuff.

Now, we must recognize that there is a similar error in use of all physical sensors as well. The art of rendering a physical phenomena such as substrate temperature of a deposition process into an arbitrary representation of volts is inherently limited by the physical approximation afforded by a conversion device, such as a thermocouple, as well as the conviction of the particular sensor designer and their particular artistic implementation.

Errors in the conversion of a physical concept, such as temperature, into a numerical representation that is to be manipulated and stored are thus invisible in the data, and only known by the physical sensor and acquisition and conversion electronics designer. This error can distort the data, often so much that incorrect conclusions about the information meaning is drawn. Fortunately, the worst-case accuracy and precision of the information can be calculated by techniques such as those described by P. H. Garrett of University of Cincinnati. These error calculations serve to limit the significance of the data, thus providing a method of specifying "noise" in the data. This technique of instrumentation design has also been used to adaptively interpret data streams in real-time process data, such that control actuation would be limited by error interpretation.

Nevertheless, the information, when converted, has been modified. To reduce the effects of measurement subjugation, the description of the actual sensor physics, dynamics, the process physics, and the method of measurement are required. An increase in apriori information will improve the accuracy of the computational representation. Increases in information representation, interpretation, accuracy, and precision will ultimately limit the development of in-situ process control and materials research.

The research achievements of this year are divided into three topic areas that investigate the indicated process sensors and how they relate to the generation of diamond like carbon material form graphite targets. The sections are Process Laser Induced Plasma Analysis, Process Dynamical Analysis, and Spatial Plume Studies. All three topics directly pertain to the development of virtual materials research, providing enhanced process understanding, sensor perception of the process, and subsequent material effects as indicated on process sensors.

Section 4a. Process laser induced plasma analysis technique design

Since MSPLD films could be made of multiple layers, an additional Air Force need could be met if multiple layer films could be made of optical grade. In addition, some method of understanding what exactly was sensed when utilizing laser induced plasma analysis was considered important. A better understanding of the plasma would be of interest for coating plastic optics with a tough exterior coating to reduce scratching and enable lightweight optical components for use in high-scratch environments, such as sand. Current diamond coatings on optical components are performed with CVD deposition under high temperatures. High temperature optical diamond coatings are currently being used on checkout register scanning laser windows, as well as military radomes and protective IR seeker windows. Due to the high temperature requirements of CVD deposition, applications are limited to optical substrates that can withstand the 700-1000°C temperatures required to grow CVD diamond. Also, since CVD diamond is "grown" and not deposited, it is difficult to achieve a uniform crystal coating primarily due to the effects of the required ": seed" crystals. A more complete understanding of what laser induced fluorescence sensors indicate is needed.

Furthermore, reducing the high substrate deposition temperature, as well as "depositing" diamond instead of "growing" films, would greatly expand the possible military, as well as commercial applications for protective diamond coatings, including polymer surfaces. This research is focused on achieving process understanding and control techniques that enable low temperature diamond deposition. Investigation of the process is accomplished by sensing methods used previously, namely, laser deposition optical emission fluorescence laser induced spectroscopy.

Since pulsed excimer laser deposition of graphite targets is known to generate the highest hardness DLC films, and is capable of low temperature high density plasma flux, this research has focused on laser deposition plasma induced fluorescence as a likely method of indicating the deposition of low temperature diamond. As found in previous research on DLC films for tribology, increasing plasma species velocities and densities is known to increase sp3 bonds, and likewise DLC film hardness. It is conjectured that a laser plume of mostly highly ionized species of high density would ultimately enable deposition of pure sp3 bond material, making the deposition of crystalline diamond films a distinct possibility.

Using the indicated laser fluorescence, various methods were tried to increase the ionized species density and reduce the quantity of lower energy and particulate plume constituents. Experiments utilizing capacitance discharge into the plasma plume had previously shown increased ion density as well as unique conical-shaped plumes as per indicated by the laser induced plasma fluorescence. Unfortunately, it was determined that the discharge probe was found to perhaps disturb plasma uniformity, creating non-uniform irregular deposition fields, although the structure of the PLD plume is little understood. Additionally, delivering the stored energy to the plume efficiently also presented a problem requiring further investigation. An alternative method of multiple laser pulses to be focused on the target was considered as a method of achieving higher plume density as per indicated by laser-induced fluorescence.

PLD Plasma Experimental Investigation

With the recent acquisition of an additional excimer laser at the ML, this opened up the possibility of dual laser pulse deposition. In this technique, two excimer lasers are used, and one is triggered off the other, and is separated by a short time delay. Both laser spots are focused through separate lenses onto the target surface at the same location. The laser light is incoherent, nor phase aligned, but impinges on the target at almost the same angle of incidence. The time delay between laser pulses is varied and the resulting plume intensity is monitored using a laser-induced optical emission fluorescence spectroscope on the resultant plume species. This result generated a conical plume that had a greenish color, and resembled the plume seen during the electrode discharge experiments. A photograph showing only the CIII ionized species is shown in Figure 12, depicting the conical shape:

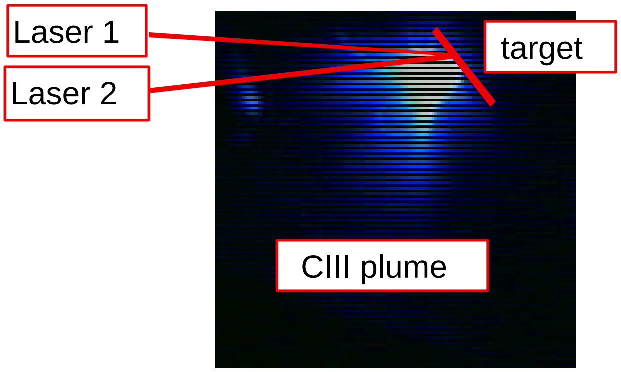

Figure 13 Double-pulse plume image at 460 nm CIII emission, showing "conical" plume

Laser induced fluorescence spectroscopy showed an increased plume emission of CIII species when multiple laser pulses were used. Investigation of the CIII emission was only performed in two locations, and the density profile of the conical plume was not investigated, although some indication can be drawn from examining the CIII emission image. This plume was similar in appearance to the plume generated by a high-energy electronic discharge. The resultant CIII emission as well as the multiple laser pulses are shown in Figure 13, showing a greatly increased emission due to the multiple laser pulses.

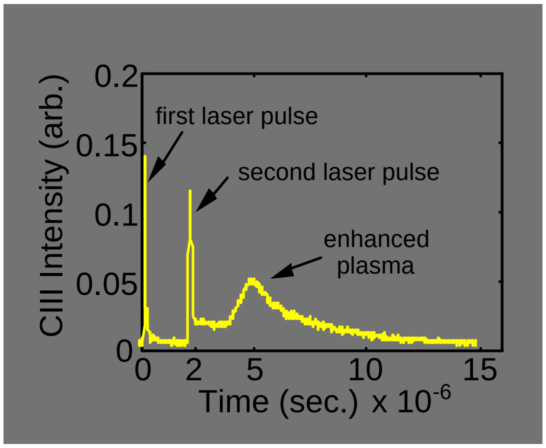

Figure 14 Multiple PLD laser pulse CIII plasma enhancement due to 2 uS laser pulse time separation.

In this case, the laser pulses were separated by 2 microseconds. It was noted that when both laser pulses were triggered simultaneously, such that both pulses arrived at the spot at the same time, the plasma emission was not maximum. This occurs perhaps because photo ablation of carbon with excimer wavelengths is not a purely photo thermal phenomena. Increased emission can be seen at about 5 microseconds after the first laser pulse impinges on the target. The laser pulses are both at 300 mJ/cm2 energy density and are both of 20 microseconds length. This increased emission was seen at the target as well as the substrate, as shown in Figure 14.

The laser pulse spikes on the laser induced plasma images were removed, for clarification, showing emission from individual laser pulses, as well as emission due to multiple pulses, showing spectral emission peak increases approximately 4 times more intense due to multiple laser pulses.

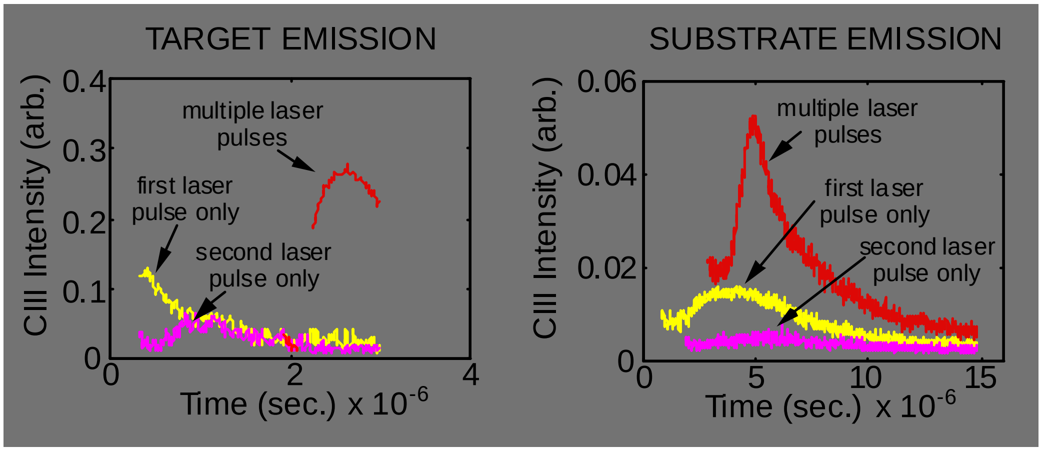

Figure 15 CIII emission enhancement due to multiple laser pulses (2 uS separation)

The temporal delay between laser pulses was also investigated, with a maximum emission of CIII occurring at 0.5 microsecond laser pulse inter-delay at the target. Increasing laser pulse separation reduced CIII emission, and increased CI emission as shown in Figure 15.

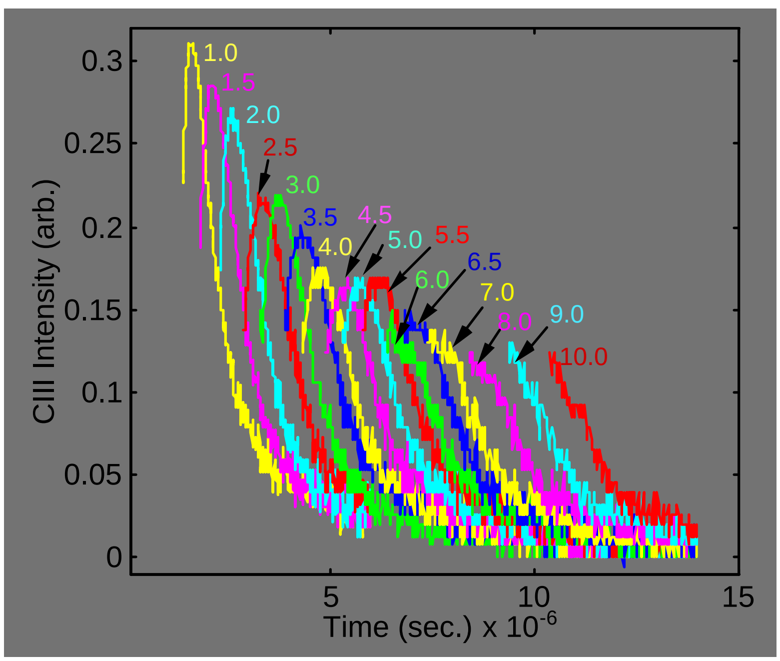

Figure 16 Increased CIII emission with dual laser pulses as a function of delay between pulses at 1 cm. Distance from the target on plume center.

Maximum CIII emission could be achieved with the delay between laser pulses of 0.5 microsecond. As mentioned previously, the laser intensity was quite less when laser pulses had no delay but arrived at the target at the same time.

Additional investigation of the CI emission was also performed with respect to delay between laser pulses. A maximum ion intensity due to the double laser pulses occurred not at 0.5 microsecond, as with the CIII species, but at 2 microsecond time separation. This is shown in Figure 16.

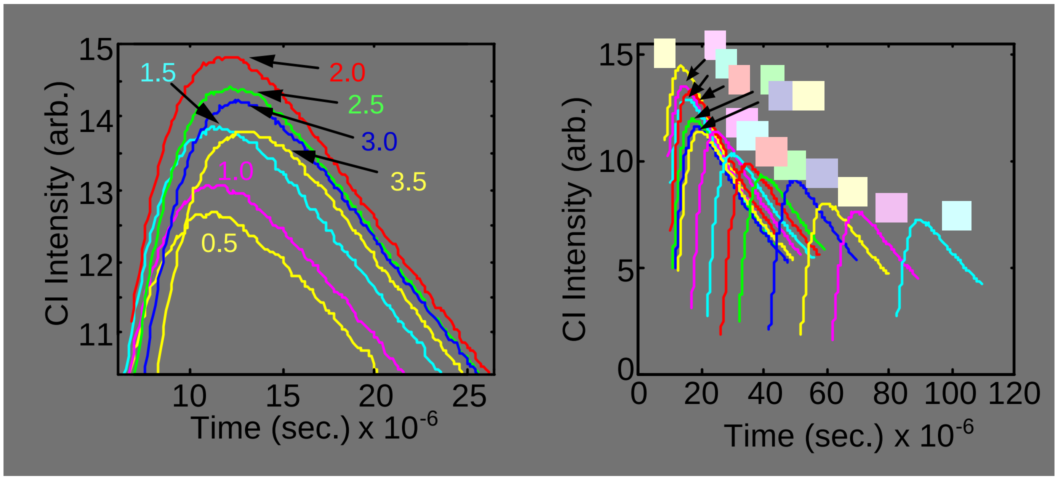

Figure 17 Increased CI emission with dual laser pulses as a function of delay between pulses.

The maximum increase in plasma intensity at an inter-pulse delay of 2 microseconds for CI species, and 0.5 microseconds for CIII species show that different plume ion species can be selectively energized. Based on these studies, the effects of utilizing multiple laser pulses on the target showed increase in the laser induced fluorescence indicated by the spectroscopes, showing that the spectroscopic data is indicating the CIII species in the plume. The increase in species can be observed relatively utilizing the spectral emission of the laser-induced fluorescence. This is necessary to utilize the laser induced fluorescence spectroscope as a control sensor to control film hardness and structure.

PLD Plasma Experimental Discussion

Utilizing this sensor data, the generation of a high density of CIII in the plasma plume could be accomplished by increasing the laser repetition rate to 2 MHz, if that were possible. Unfortunately, current commercially available pulsed excimer has maximum pulse repetition rates in the hundreds of hertz. A specialized laser would have to be built to generate bursts of this high repetition rate. It would then be possible to energize the plasma plume to a constant high CIII density, enhancing sp3 bond deposition, and possibly providing a high enough plume energy to facilitate the growth of crystalline diamond film deposition. Another option might be the enhancement of plasma deposition by using microwave energy techniques similar to those used in microwave CVD or by energy discharge techniques. Besides this, the use of optical emission spectroscopic data to generate multiple layer super hard films was addressed and found to be feasible.

Section 4b. PLD Process Dynamic Control

Multiple layer thin films of less than 2 mm are deposited via mechanical manipulation of deposition processes. Interlayer gradients are typically accomplished by rotation of substrates between two or more material deposition sources. [1] Film deposition rates are maintained with limited instrumentation as well as no feedback control. Additionally, most tribological hard films deposited on relatively soft substrates need to be deposited with a buffer or interface layer in order to bond a particular film to a substrate. Often, this buffer layer is deposited by manually ramping rotation speeds or source deposition rates, increasing film variability.

This research is focused on identifying and relating observed process characteristics to resulting material chemistry and structure with the goal of controlling material characteristics for multilayer films. Specifically, suitable process parameter and sensor identification and control techniques that are required to generate multilayer diamond-like carbon (DLC)-ceramic-metal films will be addressed. DLC films deposited by pulsed laser deposition (PLD), combined with reactive gas DC magnetron deposition of titanium to create Ti-TiN-DLC or Ti-TiC-DLC material gradients will be the focus of this work. Laser induced fluorescence (LIF) spectroscopy and a quartz crystal microbalance are used for in-situ plume sensing with the desire to control the film deposition rates and resulting deposited structure.

Self Directed Control Implementation

In lieu of uncontrolled mechanical multilayer deposition methodologies, process automation and control techniques afford greater flexibility while providing more repeatable depositions. Furthermore, hybrid deposition techniques, such as pulsed laser deposition simultaneously combined with magnetron sputtering, open up possibilities of arbitrary microstructural and stoichiometric control. It is also important to note that hybrid deposition techniques, such as magnetron sputtering combined with pulsed laser deposition have surpassed analytical understanding. Thus, empirical materials processing by self-directed control provide a reasonable alternative to the generation of gradient heterostructural tribological thin films that possess materials properties that exceed individual material characteristics.

Self directed control relies on internal process dynamics that are measured by in-situ sensing methods. Process sensors and actuators are chosen that will represent the process dynamics. In the case of hybrid processes that utilize PLD, laser energy and pulse repetition rate are chosen to actuate the deposition of a material. Film deposition is sensed by a quartz crystal microbalance (QCM) thickness sensor and laser induced fluorescence (LIF) optical emission spectroscope, (OES) as shown in Figure 17.

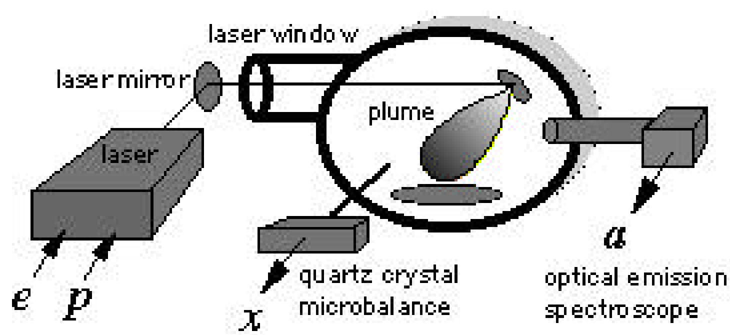

Figure 18 Conceptual diagram of desired pulsed laser deposition model.

Figure 18 is the conceptual diagram of desired pulsed laser deposition model, indicating laser energy (e) and pulse repetition rate, (p) and how they affect film thickness (x) and spectral intensity (a).

Once the sensors and actuators are chosen, an empirical dynamical model form can be chosen. The model form is based on familiarity with the process, as well as expertise available from process operators and literature. In the case of hybrid PLD, a dynamical process form such as:

$$\begin{bmatrix} \dot{x} \\ \dot{a} \end{bmatrix} = \begin{bmatrix} f_1(x,a,e,p) \\ f_2(x,a,e,p) \end{bmatrix}$$

is proposed, where:

x - Sensed quartz crystal microbalance film thickness (Å)
a - Sensed laser induced fluorescence optical emission spectroscopic intensity (a.u.)
e - Actuated laser energy density (J/cm^2)
p - Actuated laser pulse repetition rate (Hz)

Feedback objectives are then based on these in-situ measurements, and captured in the form of this dynamic model. Possible techniques such as [1] are excellent candidates to capture nonlinear time varying relationships that exist between sensors and actuators. Process in-situ sensors are also chosen or designed, such that materials characteristics are directly determined by the sensed data.

This data, combined with process and material behavioral understanding, leads to control that is directly material-focused, instead of process parameter focused. This requires possibly a complete change of perspective on the process, requiring change of control objectives, controller re-design, or process "re-modeling", since now the controller objectives are actually the desired material objectives. Self-directed control objectives are expanded to reflect product material characteristics, not the process parameters that determine them. Thus, close scrutiny of in-situ sensors become key to successful self-directed control.

Studies of PLD DLC thin films (<100 nm) have revealed a dependence of laser fluence and target material to the deposited thin film sp3/sp2 ratio. [2] In this work process control is extended to produce metal-ceramic-DLC gradient materials approaching monolayer thicknesses and incorporate them into multilayer structured coatings. Empirical process models in the form of recipes are found that manipulate the PLD process to generate multilayer films of desired composition and structure. This creates a potential for producing a new class of multiple layer gradient materials.

Description of the apparatus, in-situ sensor data interpretation, and process control strategies used, and deposition of multiple layer films using these strategies is presented.

Experimental Instrumentation Configuration

Computer instrumentation is used to affect process automation of PLD, magnetron, and substrate bias and deposition background gas. A thorough description of the hybrid process deposition techniques and apparatus are described in [2]. Additionally, the material analysis, properties, and characteristics resulting from these techniques and apparatus are described in [3],[4], and will not be discussed here. This paper focuses on the development of adequate in-situ sensing methods that would be capable of controlling a hybrid multilayer deposition process that utilizes multiple deposition techniques, so that arbitrary multilayer films can be generated by self-directed control.

In this particular study, laser deposition parameters, magnetron deposition parameters, along with substrate bias and background gas partial pressures are controlled. Several single loop feedback controllers are used to stabilize process boundary conditions, combined with in-situ sensor data to assess and control the deposition process behavior by effecting event-based control. Descriptions of the single-loop controllers that are used by events follow.

Partial pressure regulation of Ar, N_2 and C_2H_2 gas is implemented by utilizing a combination of mass-flow and pressure feedback control using hot wire and baratron gage sensing, enabling partial and total pressure control to 1 mT. Both mass flow and chamber pressure are used to actuate the throttle valve position and gas feed solenoids in a dual PI loop technique. Partial pressure setpoints are commanded from a multilayer "recipe", enabling gas pressures from 200 mT to UHV to be obtained continuously.

Titanium DC magnetron sputtering operation utilizes proportional power regulation combined with pressure regulation described above. LIF and magnetron spectral plasma emission and quartz crystal microbalance (QCM) deposition rate are monitored in-situ to indicate desired film thickness events have occurred. Again, material deposition thickness and rate setpoints are commanded from a multilayer "recipe" of process setpoints, allowing automatic control of Ti-C and Ti-C-N ratios by simultaneous operation of PLD deposition and magnetron sputtering.

Pulsed laser deposition of DLC uses a 248 nm excimer laser with focused intensities of 109 w/cm^2 depositing DLC films of high hardness. [5,6] The pulsed laser beam is focused onto a graphite target. Graphite is ablated and deposits on a substrate. Laser pulse repetition rate and energy density are actuated to deposit high-energy carbon ions and neutrals required for DLC. The laser spot is rastered across the target surface and the target is rotated to provide uniform target ablation. Laser energy stabilization is via PIN junction UV optical detector combined with laser induced fluorescence spectroscopy. Laser operating parameters are also controlled via the process "recipe" setpoints.

Multilayer Control Strategies

In lieu of complex control strategies where the entire system is consolidated as a complete dynamical system, multilayer control design was based on individual control loops operated from an event or time based schedule, as a multilayer “recipe”. The combination of these individual control loops allow for film generation ranging from Ti, to stoichiometric TiC, to DLC as described in [4].

Determining control strategies useful for generating metal-ceramic multiple layer thin films for deposition on steel substrates requires investigation of the combination of magnetron sputtering of titanium with pulsed laser deposition (PLD) of diamond-like carbon (DLC) in a hybrid fashion. Initial studies involved investigation of deposition rates and resulting material characteristics. Each process in-situ sensor data was understood independently, before hybrid control of both deposition methods was performed.

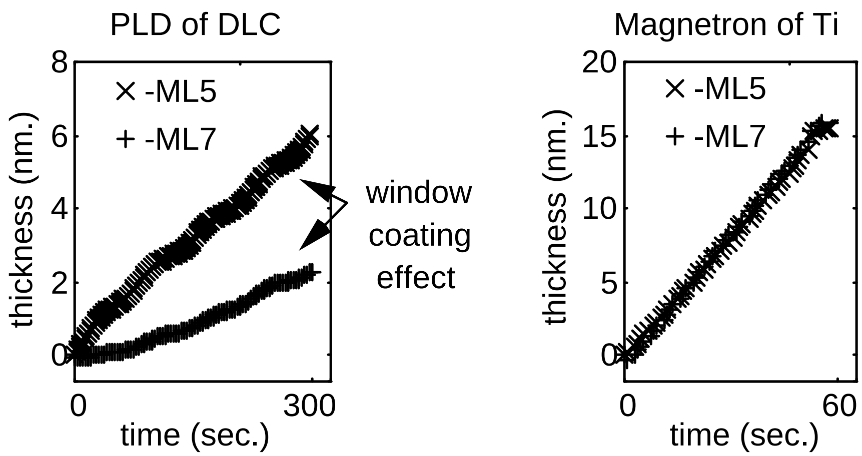

Figure 19 In-situ film thickness data for PLD of DLC (left) and magnetron sputtering of Ti (right)

Shown above is the in-situ film thickness data for PLD of DLC (left) and magnetron sputtering of Ti (right), comparing two runs made without cleaning the laser entrance window.

In situ sensing of deposition rates for both laser deposition and magnetron sputtering was determined using a QCM. Thickness data was collected over several runs. Shown in Figure 19 is a comparison of PLD (left) and magnetron sputtering deposition rates (right) for two runs (ML5 and ML7).

As can be seen from Figure 19, PLD of carbon is not at all like that of dicalcogenides, such as MoS2, requiring multidimensional dynamical control to obtain consistent material and deposition rates [7],[8], but deposited film thickness can be approximated directly from pulse repetition rate and laser energies as long as species energy is adequately high. A simple mathematical representation can then be used to estimate thickness based on laser parameters and time.

Long term dynamics due to laser beam entrance window coating was not considered, as typical deposition practice is to clean the window after every multilayer process run. Also shown in Figure 19 for PLD is a summed periodic component due to target laser beam rastering. Minimum dynamics are evident in the deposition rates of both PLD DLC and magnetron deposited Ti, with reduction of PLD deposition rate due to long-term window coating.

Once bulk thickness deposition rate stability was determined to be satisfactory, laser deposition plasma species energy would need to be determined to control the generation of DLC. This is done by utilizing LIF spectroscopy.

Spectroscope Design

Assuring adequate PLD carbon species velocities and ion densities is imperative to generating DLC as well as maintaining uniform stoichiometric TiC when the Ti magnetron is operating simultaneously with carbon PLD. Thus, in-situ sensing of carbon species velocities and intensities becomes key to depositing a uniform gradient from Ti to C using PLD and magnetron sputtering. A dual channel high speed LIF spectroscope was designed [9] that would simultaneously collect emission from two spots of the laser plume, thus permitting monitoring of species intensities and velocities at the target as well as at the substrate. A standard grating spectrometer was also used to monitor magnetron Ti emission at 400 nm.

Two LIF spectroscopes were constructed, each utilizing a 31.75 cm long 0.79375 cm diameter tube to collimate light from the plume and reduce laser light reflections from the initial flash created by the laser light impinging on the target surface. The tubes were fixed at 2 and 5.65125 cm. from the target center respectively, and placed in a line axial with the target center rotation. Optical shielding was used both inside and outside the ultrahigh vacuum chamber and measurements were made for light leaks and collimating efficiency, with less than 1% of non-collimated light leakage, and 10% crosstalk between each spectroscope channel. The optical crosstalk was primarily due to both collimating tubes sharing a single vacuum port window into the UHV chamber. The collimated light was passed through the vacuum port to two 2.54 cm. diameter spectral line filters and then into a 2.54 cm. end view photomultiplier tube. A cluster of plasma carbon (CIII) spectral lines around 460 nm was used.

Photomultiplier tube output was digitized by a dual channel HP54502 400 Ms/sec oscilloscope that was triggered by a PIN diode placed at a location where laser light enters the vacuum chamber. The laser light pulse triggers the PIN diode, causing digitization of oscilloscope waveforms from the two photomultiplier tubes. Equal cable types and lengths were used for the two spectroscope channels as well as the PIN diode to assure that no differential delays would be present due to cable velocity factors or circuit delays. Waveforms were then digitized and analyzed as well as used for sensor input to various control compensation techniques.

As expected, species intensities and velocities traveling from the target to substrate are reduced in intensity, delayed and expanded in time, but maintain a similar velocity distribution profile, with some leading edge distortion. In addition, the increased pulsed laser energy intensity detected at the target correlates with intensity detected at the substrate. Based on the species travel times, feedback compensation of the plasma could be used to control DLC material stoichiometry.

Unfortunately, it was found that stable plasma emission at the target did not always generate uniform stoichiometric films. There was also an effect that was due to different laser beam rastering techniques. Additionally it was found that compensation based on a single spectroscope tube location needed constant re-calibration and re-tuning when substrates of different sizes and shapes were used.

Depending on the spot location at the target surface, widely different waveforms and species velocities were found at the target and substrate that were highly irregular, and similar to waveforms seen in [10]. Only when the laser spot was centered on the target did the LIF substrate waveform correspond to the target in intensity and waveform shape. Furthermore, these LIF waveform irregularities were difficult to predict. A controller that relied on the LIF waveform close to the target would have difficulty in reliably controlling substrate species velocities and intensities.

The use of LIF waveforms as in-situ sensors require careful consideration of the LIF data with respect to sensor location, raster pattern, and other factors. Using LIF as an in-situ sensor for stabilization of PLD, species during co-operation of magnetron sputtering would be difficult unless care is given to the implementation. Furthermore, compensator design based on LIF waveform time characteristics must be capable of interpreting PLD parameters, such as laser spot location and substrate height before generating uniform PLD DLC in multilayer films. Otherwise, an average LIF waveform can be used, with subsequent loss of control resolution in the multilayer stack.

Implementation of self-directed control requires careful consideration of in-situ sensor data with respect to all process parameters that affect material attributes. A reduction of controller complexity will result with in-situ sensors that adequately reflect material characteristics. Otherwise, it becomes difficult to correlate many parameters with in-situ data, ultimately increasing the required controller complexity. Thus, it becomes imperative to understand sensor data and process effects, so that blunders in controller design can be avoided.

Spatial Plume Implementation

This research focuses on identifying pulsed laser deposition (PLD) of diamond as if carbon (DLC) observed plume characteristics to resulting material chemistry. Observed process parameters ultimately determine the deposited film mechanical properties. Preliminary studies on laser induced fluorescence of carbon by pulsed laser deposition showed unusual velocity profiles when the ablated material was observed with a time of flight laser induced fluorescence (LIF) optical emission spectrometer. (OES) Further investigation of spectral emission temporal characteristics was performed to reveal interaction between the substrate and laser plume, with an increase in species energy at the substrate. Species velocities and other non uniformities were also observed at the target and substrate with respect to the laser spot position on he target. Plume ion velocity and density distribution was found to be asymmetrical and non uniform, creating stratified film structures.

End Use Demands

Recent aerospace demands have focused tribological research on improving wear resistant materials. [11] Pulsed laser deposition (PLD) is a promising deposition technique for generating diamond like carbon films [12] of high hardness. [13],[14] Additionally, high species kinetic energies combined with the high instantaneous flux of PLD have been able to create diamond like carbon films capable of structural transformation. [15] Utilizing laser induced fluorescence (LIF) optical emission spectrometry (OES) as a process sensor; it has been found that film uniformity is dependent upon process parameters. Furthermore, uniform film structure and deposition rates across wide areas are required to generate films sufficient for optical components. Precisely controlling deposited film composition and structure is necessary when utilizing hybrid processes to generate multiple layer structures of gradient elemental composition. [16],[17],[18],[19] With this in mind, a more complete analysis of plume species quantity and composition is required to satisfy film uniformity demands.

Pulsed laser deposition of diamond as if carbon (DLC) utilizes a high energy pulsed excimer laser. A laser pulse of 20 nS duration is focused on a 1” diameter graphite target with intensities of 1 J/cm2 to create a high-energy plasma plume. This plasma plume is generated in an ultrahigh vacuum environment (10-6 Torr) and contains carbon neutrals and ionized species. The ionized plasma plume accelerates away from the target and impinges on a substrate to be coated. The plume interacts with the substrate and creates unique film structures and properties. Instrumentation of this deposition process is desired so that the deposition method can be more fully understood. This report focuses on how process parameters affect the deposited carbon film and how these parameters relate to optical emission characteristics.

OES Construction and Implementation

A dual channel spectral line laser induced fluorescence (LIF) optical emission spectrometer (OES) was designed that would simultaneously collect emission from two spots of the laser plume for one or two different ionized species. [20] This was accomplished by designing two high-speed spectroscopes and mounting both on the PLD vacuum chamber. The apparatus is shown in Figure 20.

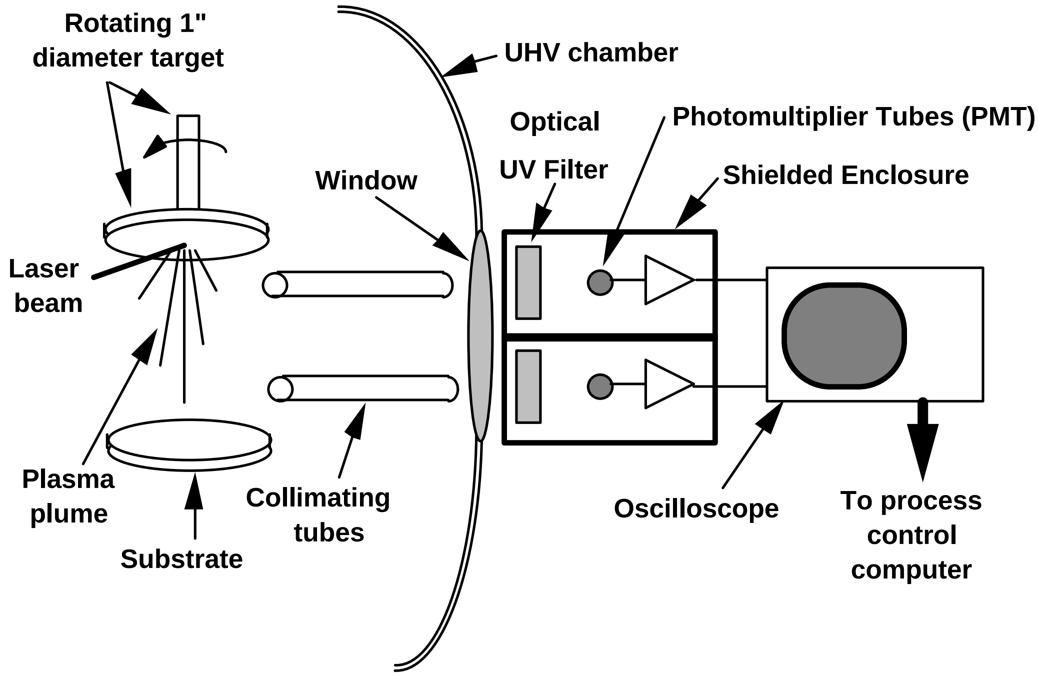

Figure 20 Dual channel laser induced fluorescence(LIF) optical emission spectroscopy (OES) apparatus used in these experiments.

Two laser induced fluorescence spectroscopes were constructed, each utilizing a 31.75 cm long 0.79375 cm diameter tube to collimate light from the plume and reduce laser light reflections from the initial flash created by the laser light impinging on the target surface. The tubes were fixed at 2 and 5.65125 cm from the target center respectively, and placed in a line axial with the target center rotation. Optical shielding was used both inside and outside the ultrahigh vacuum chamber and measurements were made for light leaks and collimating efficiency, with less than 1% of non collimated light leakage, and 1% cross talk between each spectroscope channel by the use of a dual window custom UHV flange. Earlier prototype LIF OES had difficulty with optical cross talk due to both collimating tubes sharing a single vacuum port window into the ultrahigh vacuum chamber. The collimated light was passed through the vacuum port to two 2.54 cm diameter spectral line filters and then into optically isolated 2.54 cm end view photo-multiplier tubes.

Photo-multiplier tube output circuitry is impedance matched to reduce signal distortion, and digitized by a HP50000 series 500 Ms/sec oscilloscope. Oscilloscope trigger is generated by a PIN diode placed at a location where laser light enters the vacuum chamber. The laser light pulse triggers the PIN diode, causing digital storage of oscilloscope waveforms from the two photo-multiplier tubes to begin. Equal cable types and lengths were used for the two spectroscope channels as well as the PIN diode to assure that no differential delays would be present due to cable velocity factors. Integral PMT high voltage power supply modules were used, with adjustable high voltage to reduce PMT overload. Although this was somewhat effective, the PMT must be operated in a quasi linear fashion. Reducing PMT anode voltage to below 400 volts caused final dynode saturation and nonlinear optical input versus voltage output transfer function.

Digitized waveforms were captured in a process control computer, along with other process sensor data. This data was analyzed and time based plots were generated.

Laser Energy Effects On Plume Emission Intensity

A comparison of OES temporal emission at the target and substrate are shown in Figure 21. The plots shown were made of the intensities with respect to time. Effects due to changing laser energy agree with wave forms found in Harilal. [21] The waveforms shown here were collected at a laser pulse rate of 10 Hz. Effects from increasing laser energy increase ion emission intensities and species velocity at both the target and substrate. Corresponding intensity at the substrate is delayed due to transit time of the ionized species from target to substrate. Mean species velocity is approximately 5 km/sec, and show a decrease in mean intensity of approximately 3.75.

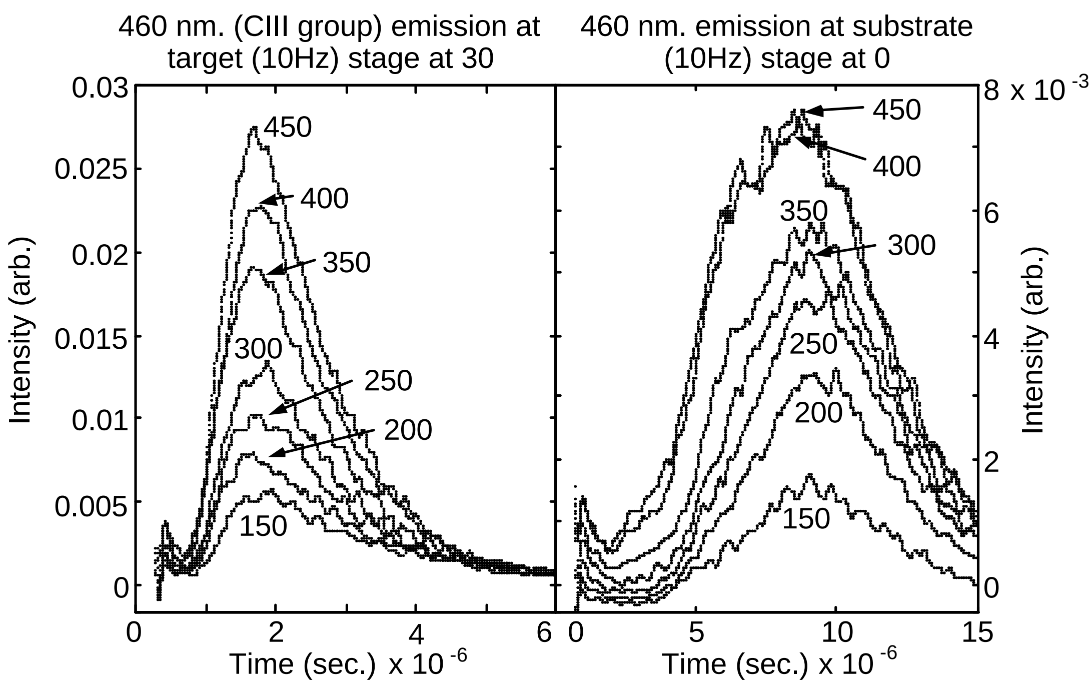

Figure 21 Dual channel LIF OES showing laser energy effects at both the target and substrate.

Although, as indicated above, much energy is lost in the traverse of the material, there is still sufficient energy to generate an intense plasma at the surface to be coated. In this way, it is possible to generate hard materials with unique structures without heating the surface to be coated.

Substrate Position Effects On Plume Emission Intensity

The effects of substrate position on species emission were observed. In these experiments, the substrate height was varied to observe the ion species intensity in the substrate vicinity with a single OES collection tube. The plots shown were made of the intensities with respect to time. Substrate height was changed from 0, 2, 5, 10, 20, and 30 mm from the collimating tube center and species emission intensity was collected. Two emission lines were collected, one at 780 nm, corresponding to singly ionized carbon, and then another at 460 nm, corresponding to triply ionized carbon. The change in spectral emission wavelength was done by changing the spectral line filter. The laser energy and pulse rate were held constant, at 400 mJ/cm2 and 10 Hz. Plots of target emission showed no change due to substrate height, and are not shown.

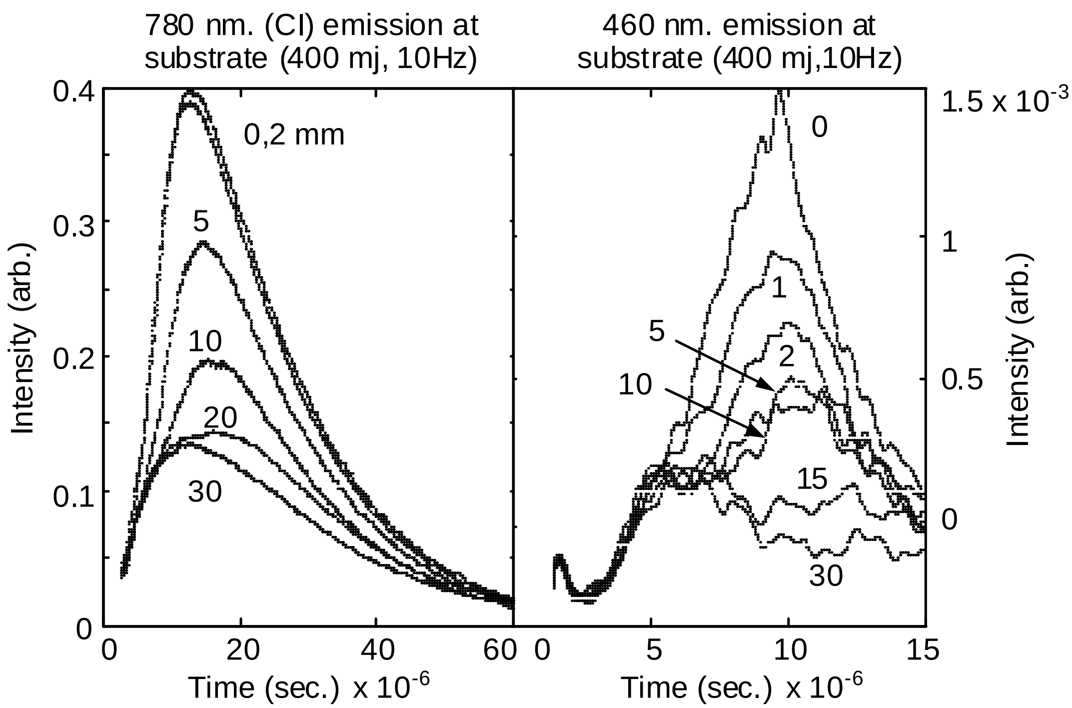

Figure 22 Dual channel LIF OES showing effects of varying the substrate position at two spectral lines for a constant laser energy and pulse repetition rate.

As can be seen in Figure 22, waveforms due to the 460 nm emission were weak in intensity as compared to the singly ionized 780 nm carbon, but increased intensity in the vicinity of the substrate at both wavelengths. This increase suggests an ionization of the species as they come in contact with the substrate. This substrate ionization effect suggests that PLD plume species are not direct line-of-sight deposition, but dispersive, due to the ionization of the carbon at the substrate. A fine wire mesh was placed directly over the substrate and a film was deposited. The deposited film showed minimum masking due to the wire mesh. A SEM of the masked surface is shown in Figure 23.

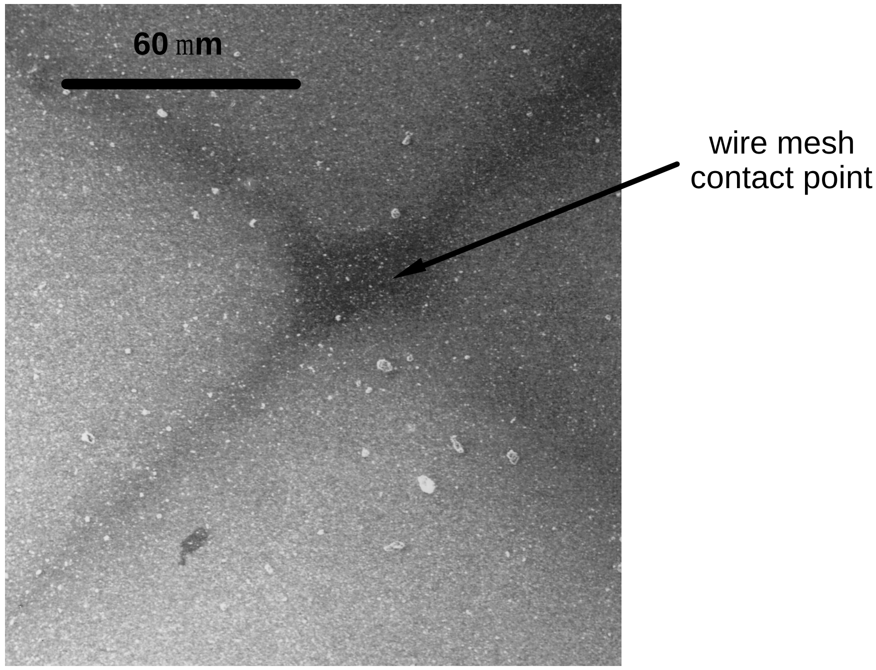

Figure 23 Surface SEM of film masked with wire screen.

As can be seen from this micrograph, the process does not exhibit a line of sight coating mechanism. The high species velocity releases the kinetic energy heating the surface to be coated, affecting the overall deposition rate as well as surface material structure. It would be oversimplification to assume this as the only surface interaction mechanism, though.

Laser Target Spot Location Effects On Plume Emission Intensity

Next, laser energy and pulse rate were held constant, and the laser spot was moved vertically on the 1” diameter target while spectral emission was collected from a fixed tube position. The plots shown were made of the intensities with respect to time. The laser spot on the target surface is approximately one square millimeter in size, and was placed at -12.5, -6.5, 0, 6.5 and 12.5 m from target center along the vertical axis. Emission at the substrate was collected for 460 nm as well as 786 nm. These are shown in Figure 24 for four different target spot locations.

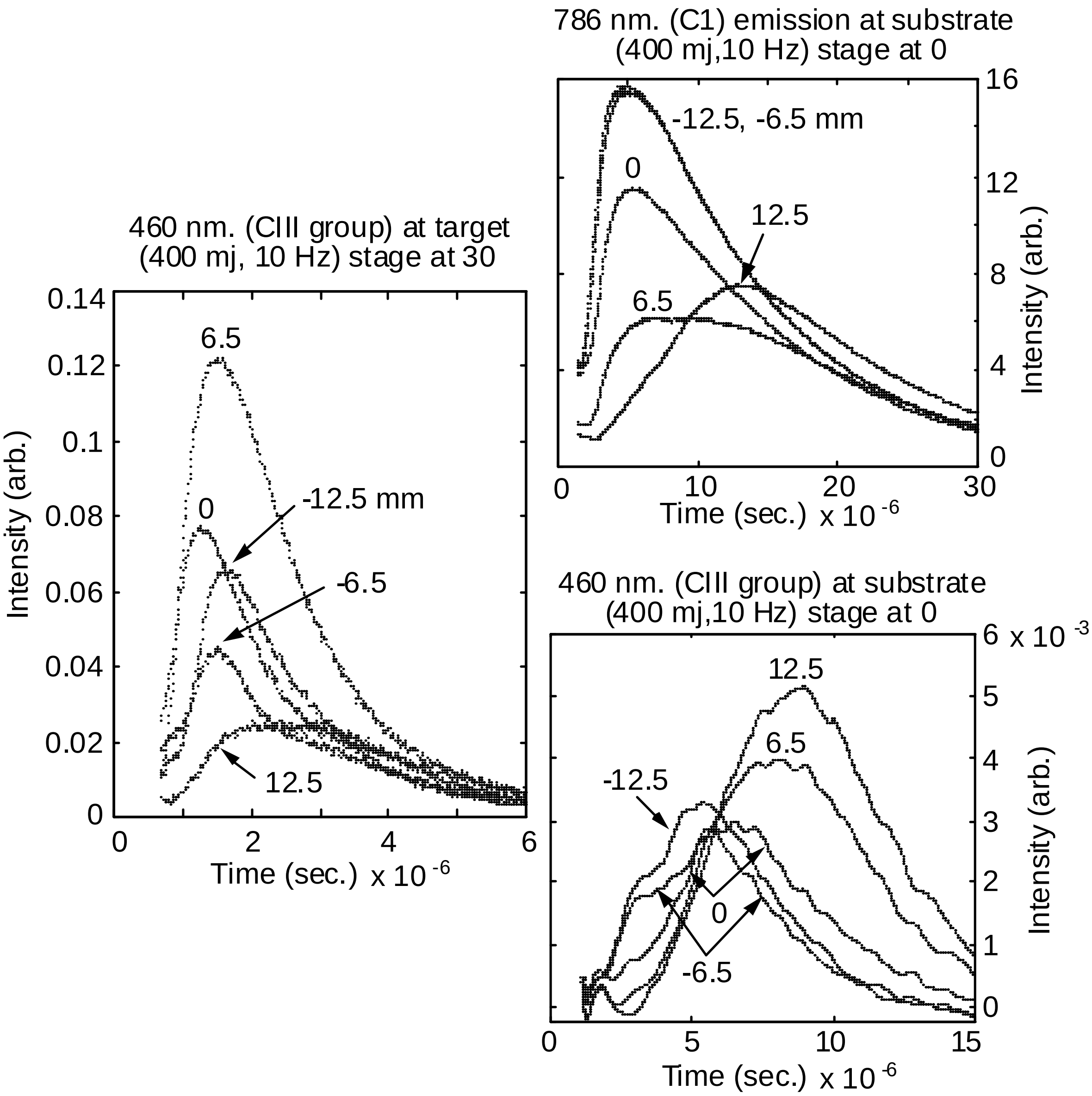

Figure 24 Dual channel LIF OES showing effects of varying the spot position on the target as well as two spectral lines on the substrate for a constant laser energy and pulse repetition rate.

Emission intensities at the target due to spot location do not directly correspond to emission intensities at the substrate, indicative of plume ion non uniformities, or perhaps plume chemistry. The unusual shapes correspond to those seen in Harilal [23] as well as reported in Voevodin. [18] Substrate emission intensities from the 460 and 786 nm spectral lines also do not correspond to spot location, suggesting that spectral ion ratios in the plume are not constant, as well as no uniform. These plume non-uniformities may be due to target surface roughness or laser spot non-uniformities.

Integrated Plume Emission Intensity

A spatial intensity image was captured of 460 nm emission intensity. In an attempt to understand the erratic spectroscope waveforms, sensing spatial distribution of the CIII carbon species was necessary.

Instead of time based species intensities of the LIF spectroscope, a spectral line image camera was constructed to determine spectral intensities of the CIII carbon ions in a spatial sense. This was done by removing the PMT in the LIF spectroscope, and installing a CCD camera array. The video image was then digitized and stored in the computer for signal enhancement and post analysis. The results of the image camera are shown in Figure 25 for CIII ions, with the target and substrate positions drawn on the intensity plot.

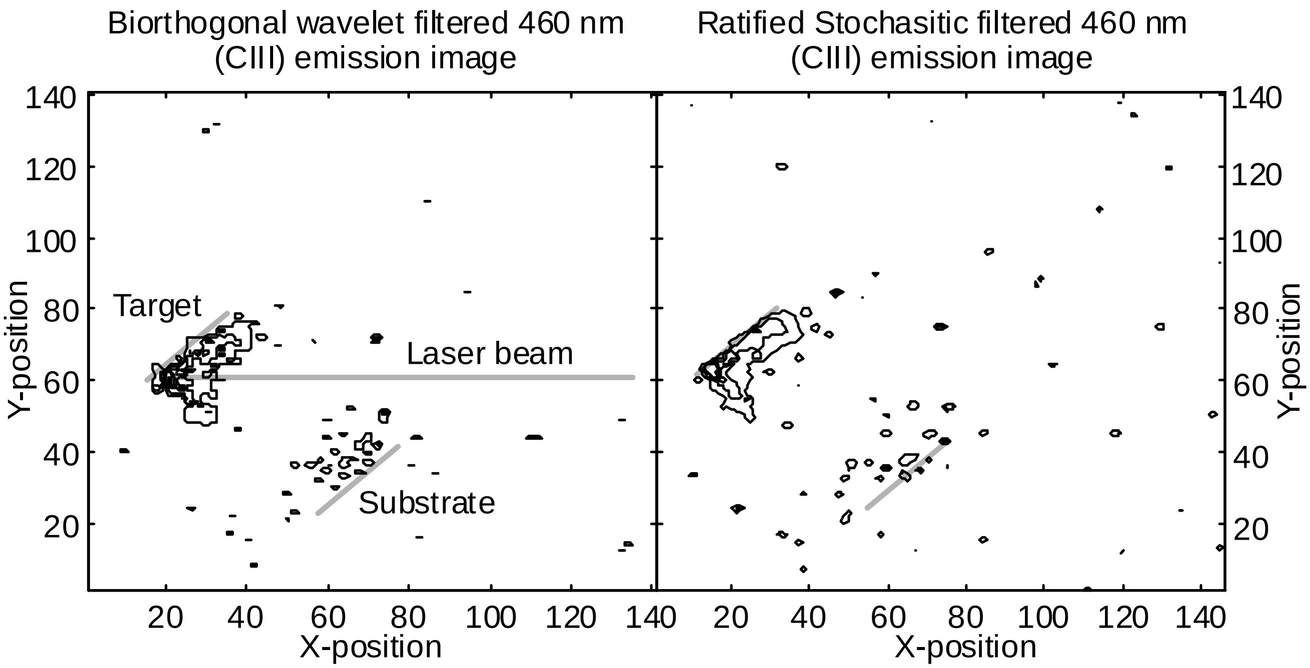

Figure 25 Carbon CIII images of the target and substrate.

Shown above are carbon CIII images of the target and substrate using two different image enhancement techniques (Wavelet and Ratified stochastic filters), showing integrated ion intensity distribution due to 10 laser shots.

Due to the relatively weak CIII emission, several laser shots were integrated to generate the contours shown in Figure 25. Post image processing was also used to enhance the images, including biorthogonal wavelet filtering and ratified stochastic techniques. As expected, CIII emission intensity is greatest at the laser spot location on the target illumination of the total target surface by laser induced CIII species can also be seen, possibly causing the asymmetrical distribution of plume species densities due to columbic expansion effects. Also observed is re-ionization of carbon CIII species at the substrate. This ionization indicates that different shaped substrates affect the detected LIF waveforms at the substrate. It was also noted that the irregular shape of target-emitted species would cause irregular spectral emission at the substrate.

Laser Beam Scanning Film Morphology Dependence

As described previously, PLD utilizes a laser beam that is focused onto a target. This spot is usually scanned, or "rastered" across the target surface. This rastering is necessary to avoid excessive target erosion that would occur if the laser beam was left focused in a single spot on the target surface.

Additionally, the target is rotated. Some raster patterns move the spot in a strictly horizontal or vertical scan on the target surface, but in the case of these experiments, a two dimensional raster is used to further minimize target erosion. This two dimensional raster pattern is accomplished with a two axis mirror controller that moves the laser spot on the target to generate a complex raster pattern consisting of interlaced horizontal lines as shown in Figure 26.

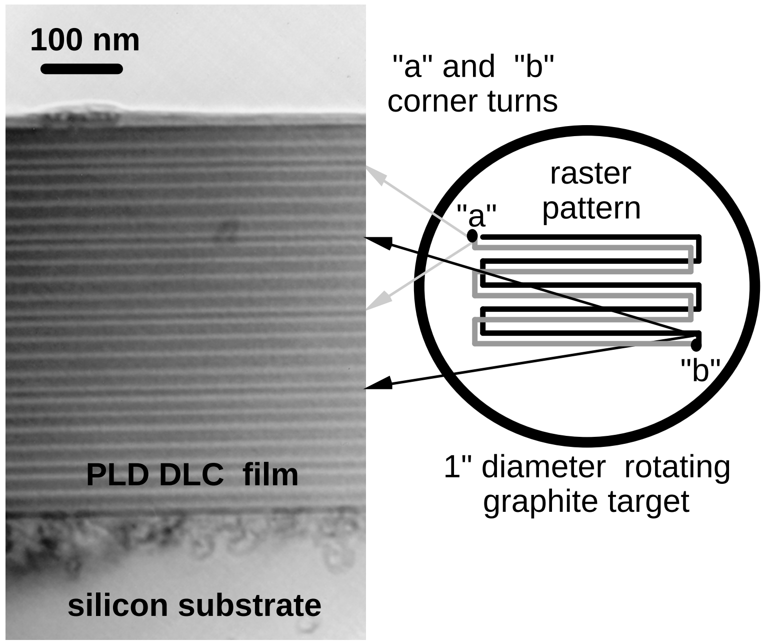

Figure 26 Cross-sectional TEM of PLD DLC film, indicating periodic density variations corresponding to target rastering of PLD laser beam.

The plume spot location non-uniformities observed with LIF OES suggests that plume ion densities are not consistent throughout the plume. Previous studies have indicated that ion densities and velocities affect film density and hardness. Thus, the deposited film density should be affected by the laser spot location, which is determined by the raster pattern.

A periodic pattern is visible in the film, and indicates density changes due to the previously described plume non-uniformities. The plume flux density is higher in the central regions, and rapidly decreases with the angle from the normal to the target surface. These cause the film density variations, when the plume is scanned across the substrate surface, following laser beam scanning on the target surface. Due to the raster spot location, the film is periodic in cross sectional morphology, caused by density variations. This is clearly seen in cross-section TEM photograph and corresponding graphic depicting the raster pattern on the target surface is shown in Figure 26.

A cross sectional TEM of a PLD deposited DLC film shows indication of periodic density variation, as described in Walck. [22] A close examination of the laser spot rastering pattern on the target shows a strong correlation to the periodic density variation. The period corresponds with the raster pattern used, including decreased periods due to raster pattern “corner turns” necessary to generate the interlaced laser beam raster scans.

OES Results And Discussion

Good success has been achieved by utilizing process control to deposit films using PLD with other deposition process, such as magnetron sputtering. Although stoichiometric control was achieved by simultaneously depositing two different materials, the precision in stoichiometric control is ultimately limited by plume ion intensity non-uniformities. The periodic film structures also make it difficult to deposit films for optical applications, such as optical filters.

Alleviating stratified film structures by mechanical means rationalizes the current process techniques of mechanically rotating the PLD substrate [23] to obtain more uniform films. This mechanical means to alleviate the effects of plume non-uniformities probably only changes the periodicity of the density variations, because it does nothing to reduce the plume non-uniformities. Further, it becomes impractical to rotate parts of complex geometry and shape, while still obtaining a uniform coating over the part surface. An additional sensing method is needed [24], so that implementation of precise control can be made possible. [25]

Section 4c. MSPLD Experimental Feedback Control Results

Current research efforts are focused on the processing of and accurately controlling multilayer depositions of diamond like carbon (DLC) films. Of prerequisite importance is the ability to reliably produce DLC films that exhibit desired hardnesses and uniformity, thus requiring deposition at low (<200°C) part temperatures. It is also necessary to control film stress so that DLC can be "graded" into TiC or Ti parts without high stress boundaries. Magnetron sputtered Ti and TiC is currently a well established deposition technique, and can generate films of desired hardnesses in a repeatable fashion. Thus, it is not necessary to further develop magnetron process control technology, but integrate magnetron sputtering with other available methods.

Uniform DLC films containing high sp^3/sp^2 ratios are known to exhibit superior hardness but are difficult to deposit reliably at low temperatures and thicknesses exceeding 300 angstroms. High hardness alone is not enough to meet DLC tribological applications. Bulk film stress must also be contained within reasonable bounds to obtain desired film adhesion, and to prevent catastrophic film disintegration.

One method of controlling film hardness is by introducing interstitial layers of TiC between DLC layers by magnetron sputtering. High interface stress is also present at the substrate/film interface as well. A TiC layer can also be used to relieve stress of a DLC film on a Ti substrate. Thus, a multilayer film can be made of alternating layers of TiC and DLC on Ti substrates. The transition between TiC to DLC and back can be done in a "graded" fashion, by varying stoichiometric amounts of Ti and C. In this fashion, film stresses can be bounded, thus providing a superior film.

In order to control film stoichiometry and microstructure, process parameters that affect DLC hardness and composition must first be identified. A survey of several papers on DLC show a relationship between laser wavelength and energy density required to produce a hard DLC film.

Pulsed Laser Deposition (PLD) has been shown to be an excellent technique to deposit adherent, crystalline thin films of complex chemistry and morphology. Its primary strength is stoichiometric transfer of material. PLD is a complex, highly photo-energetic process, the physics of which are not completely understood. Several methods are available to improve the understanding of the PLD process. One method is to improve the observation technique of the process in real time through improved data acquisition tailored for process modeling applications. Another is to reduce process uncertainty, through the application of feedback control.

One method of simplifying the PLD process is to decompose it into the sub processes that are the three energy transfer regions that occur. In the first sub process, energy from the laser is transferred into material in motion. The material is motivated by the laser impinging the target surface. The energies transferred in the material in motion within the plume constitute the next sub process. Material chemistry may occur within this second sub process. The third sub process is where target material comes to rest on the substrate surface, transferring the plume energy and creating a thin film.

To gain control of the PLD process, suitable sensors must be identified and developed for *in situ* measurements of essential state variables of the deposition process. Sensor information will generate further understanding of basic film growth characteristics. The primary goal of self-directed control of PLD is to significantly improve the consistency and quality of the films, while simultaneously increasing process knowledge. Effective control of PLD also requires the identification of actuation parameters. The high-energy laser is the main source of ablation energy, and a suitable choice for the actuator. Since cavity voltage of the laser can be varied per pulse, real-time actuation of energy density can be directly changed during a deposition. Similarly, the pulse frequency can be varied in real time to affect the instantaneous energy on the target. The excimer lasers used for PLD are typically constrained to fixed pulse lengths during operation, so varying the pulse length is not currently possible.

Initial observation of actuator and sensor data indicated that PLD exhibits a nonlinear ‘dynamical’ behavior. Therefore, a nonlinear dynamical process model representation was used to describe the PLD process. Material deposition relationships were empirically found by varying the laser energy density and the pulse repetition rate and recording sensor data. Derivative information was measured over designed experimental epochs. Molybdenum disulfide (MoS2) was the target material selected for the initial empirical model development. The use of dynamical feedback control of PLD has permitted growth of deposited film crystal structures that were previously unattainable using open-loop control techniques.

The following discussion will be subdivided into two (2) projects. The first is the continued ‘in-house’ collaborative effort in PLD of tribological materials.

In situ **Process Modeling and Feedback Control Problem Statement**

This research of pulsed laser deposition (PLD) is focused on identifying and relating observed plume characteristics to resulting material chemistry and structure with the goal of controlling material characteristics for multilayer films. Laser induced fluorescence spectroscopy and a quartz crystal microbalance are used for *in situ* plume analysis. Studies of PLD diamond-like carbon (DLC) thin films (<100 nm) have already revealed a dependence of laser fluence and target material to the deposited thin film sp^3/sp^2 ratio. In this work, process control is extended to produce metal-carbide-DLC gradient materials and incorporate them into multilayer coatings. Empirical process models in the form of recipes are found that manipulate the PLD process to generate multilayer films of desired composition and structure. This creates a potential for producing a new class of materials.

Total deposited thicknesses of smooth diamond-like carbon (DLC) films of high sp3 bonding fraction and uniform structure have been traditionally limited to 0.5-1 mm final thickness. This is due to the inherent increase in the film stress vector sum, which results in inevitable film delamination [26],[27],[28]. Even when an appropriate sample preparation technique is used, delamination from the substrate occurs, due to residual stresses, hardness, and modulus differences of DLC and substrates of comparatively softer materials such as metals or steel alloys. Practical use of DLC films of relatively high thicknesses requires interposition of hard DLC layers with stress releasing elastic interlayers, such as metals. Thus, a motivation exists for depositing DLC with interlayers. In order to reduce strain at the multilayer boundaries, a stoichiometric gradient would also be useful [29]. In summary, a gradient stoichiometry multilayer film would be capable of reducing total film stresses, while still permitting relatively thick films (>1 mm.) to be deposited.

The generation of gradient stoichiometric films require a method of process control, such that film stoichiometry can be accomplished repetitively and consistently. Furthermore, simultaneously utilizing more than one deposition process method, combined with the execution of multiple steps, increases the operator load to beyond human capabilities. In order to generate multiple layer films with tens or hundreds of layers utilizing multiple deposition techniques, process control and automation becomes imperative. The below describes an apparatus and a method capable of generating multilayer films with graded interfaces utilizing ultraviolet 248 nm PLD of graphite targets combined with direct current magnetron sputtering of titanium [30].

Experimental Process Control Design

Computer instrumentation is used to affect process automation of the PLD laser pulse energy and repetition rate. Magnetron power and background gas partial pressures are also controlled, allowing more capable monitoring of film deposition process parameters. Several single loop feedback controllers are used to stabilize process boundary conditions, combined with *in situ* sensor data to assess process behavior. Partial pressure regulation of Ar gas is implemented by utilizing a combination of mass-flow control and pressure sensing by Baratron gage, allowing for gas regulation to 1 mT. Both mass flow and chamber pressure are used to actuate the throttle valve position and gas feed solenoids. Partial pressure setpoint control is then commanded from a "recipe" of process setpoints, enabling gas pressures from 200 mT to UHV to be obtained.

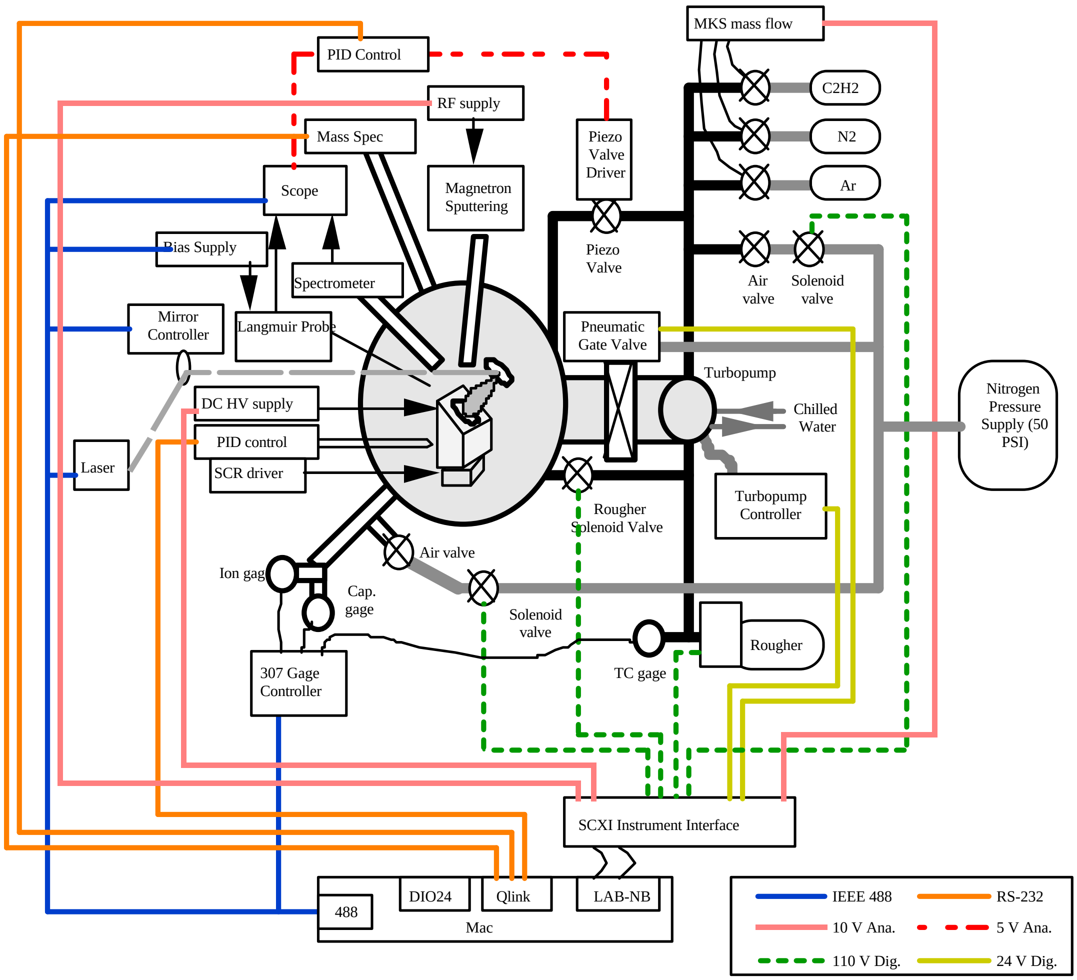

Figure 27 Preliminary prototype multilayer apparatus system diagram and instrumentation.

Titanium DC magnetron sputtering operation utilizes power regulation combined with pressure regulation described above. Spectral plasma emission and quartz crystal (QCM) deposition rate are monitored *in situ* to sense the sputtering deposition condition. Setpoint control is commanded from a "recipe" of process setpoints, allowing automatic control of Ti-C ratios by simultaneous operation of PLD deposition and magnetron sputtering. Excimer laser pulse repetition rate and energy density are actuated to deposit carbon from a rotating graphite target, allowing for film generation ranging from Ti, to stoichiometric TiC, to DLC. Laser energy stabilization is via PIN junction UV optical detector combined with laser induced fluorescence spectroscopy. Laser operating parameters are also controlled via the process "recipe" setpoints.

Apparatus Description

A diagram of the apparatus suited to create graded multilayer film in Figure 27. The apparatus utilizes computer automation and control to operate gas valves, magnetrons, mass flow rates, substrate temperature, and laser parameters. QCM and LIF spectroscopy sensors are used to provide *in situ* assessment to the process control algorithms. The apparatus and algorithms also allow for automatic setup of the chamber, and then execution of the "recipe" to generate the film.

Software that operates and controls the apparatus executes commands that control digital and analog signals that control instruments. Substrate temperature, chamber total gas pressure, and magnetron sputtering plasma emission spectroscopy are process boundary condition parameters that are controlled by independent feedback loops. These independent boundary condition control loops only require setpoint data to effect process changes.

Process Control Results and Discussion - Experiment Method

Recipe film growth has been used in other deposition processes, e.g. molecular beam epitaxy [31]. Initial investigation into combined dynamics of the PLD and magnetron deposition system indicated that internal process dynamics are greater than one second, primarily due to the PLD of carbon. These slow dynamics permit the generation of a desired film gradient by stepwise setpoint change. Slow process dynamics, combined with computer control at millisecond resolution, eliminate the need for a multi-dimension process dynamical controller.

Gas residency calculations require the computer to update setpoint values in less than 1.3 seconds for the chamber setup used. Ti-DLC and TiC-DLC thickness transitions of 5 nm were originally desired, so setpoint step-approximation permitted deposition of smooth transitions. These transitions were then used to generate the desired material stoichiometric gradients. Control techniques similar to this are referred to as "Gain-Scheduling" [32]. A plot of magnetron voltage, pressure, and spectral output, for laser cavity voltage and pulse rate are shown in the previous film transition Figure 28 for four step approximated gradients, labeled "a", "b", "c", and "d". Each recipe step is limited by the process transient time of approximately 2 seconds. Magnetron deposition rate is approximately 0.3 nm/sec, indicating an achievable minimum of 0.6 nm per film transition.

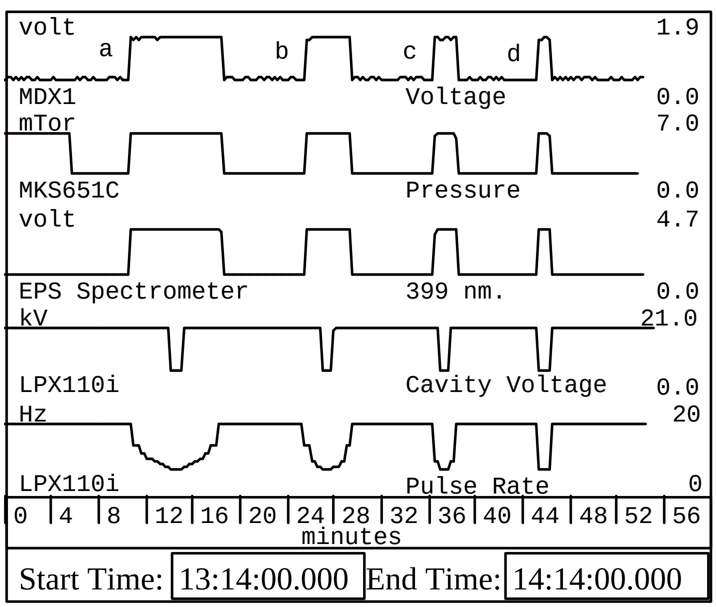

Figure 28 Process Parameter film transitions for four (4) different gradient layers

Shown next are the resultant film morphological identities corresponding to the four different gradient layers as seen by an edgewise SEM.

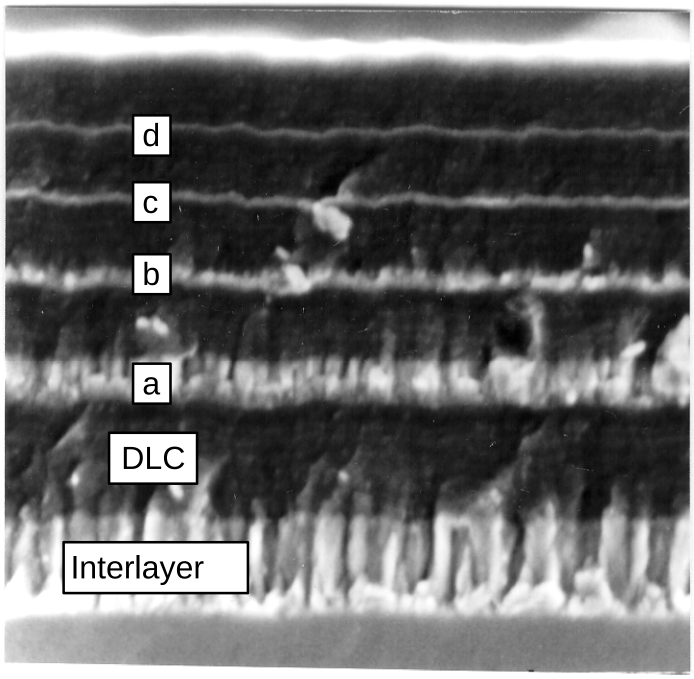

Figure 29 SEM depicting four graded multilayers generated under recipe process control

A high resolution SEM image of the resulting film generated by step approximation Figure 29. The transitions labeled "a", "b", "c", and "d" corresponds to those shown in Figure 29, with "d" being the 0.6 nm gradient. As can be seen [13] large gradients are possible that contain composition ranging from pure DLC to pure Ti. These gradients are created by a reduction of laser pulse rate over time. A decrease in laser energy density, magnetron power, argon pressure, or their combination can also be used. If recipe control is added, then films with numbers of layers and gradients over a wide range of desired thicknesses and composition are possible. Thicker, more gradual gradients can be generated by stepwise approximation to a continuous slope variable.

Each "recipe" was used to deposit a pre-designed film structure that has desired material characteristics. By combining the time-based setpoint changes over the entire deposition, generation of complex multilayers is possible, including boundary, interface, and multilayers. Since the multilayer film repeats, a looping structure was included so that an arbitrary number of multilayers could be selected from a single recipe edit field. In the case of Ti-DLC and TiC-DLC, graded films of 1-100 nm per layer are possible using this method [10]. The recipe method of control is capable of generating rate changes necessary for graded films without complex multiple dimension control algorithms. Recipe film growth assumes no internal process dynamics are occurring or are compensated for by each process boundary condition control loop. Individual boundary conditions are inherently band limited and stabilized by single loop controllers, so the process is globally stable. Consequentially, recipe methods are able to generate similar films each time, even though the process may vary.

Unmodeled Dynamic Effects

Although PLD of carbon is relatively stable when compared to PLD of dichalcogenides, [33,34] PLD dynamics of carbon become apparent over long periods of time when depositing large numbers of multilayers. [35] The dynamics are due to target ablation damage and temperature effects as well as window coating. Due to the increased process complexity, instrument faults also become more likely, altering the final film.

Interlayer recipe executed once:

HOUR	MIN.	SEC.	E (mJ)	P (Hz)	PR (mT)	B (V)	M (W)	G (U)	Result
0	15	0	0	0	100	-600	0	Ar	Etch (Si)
0	30	0	0	0	0	0	0	0	Pause
0	35	0	0	0	2	0	100	Ar	Ti 100
0	40	0	200	1	2	-60	100	Ar	Ti 90/C 10
0	41	0	200	2	2	-60	100	Ar	
0	42	0	200	3	2	-60	100	Ar	Ti 50/C 50
0	45	0	200	4	2	-60	100	Ar	
1	10	0	200	6	2	-60	100	Ar	
1	19	0	200	12	2	-60	100	Ar	Ti 25/C 75
1	20	0	200	20	0	-60	0	Ar	C 100 soft
1	25	0	0	0	2	0	100	Ar	Ti 100
1	26	0	0	0	0	0	0	0	Pause

Multilayer recipe section for Ti-TiC-DLC-[Ti-DLC]20: (ML5)

HOUR	MIN.	SEC.	E (mJ)	P (Hz)	PR (mT)	B (V)	M (W)	G (U)	Result
0	1	0	200	20	0	0	0	0	C 100 DLC
0	4	0	0	0	20	0	0	Ar	
0	5	0	0	0	2	-60	100	Ar	Ti 100

Multilayer recipe section for Ti-TiC-DLC-[TiC-DLC]20: (ML7)

HOUR	MIN.	SEC.	E (mJ)	P (Hz)	PR (mT)	B (V)	M (W)	G (U)	Result
0	1	0	200	20	0	0	0	0	C 100 DLC
0	4	0	0	0	20	0	0	Ar	
0	5	0	200	4	2	-60	100	Ar	Ti 50/C 50

Table 3 Interlayer film recipe combined with multilayer recipes used in both Ti-TiC-DLC-20x[Ti-DLC] (ML5) andTi-TiC-DLC-20x[TiC-DLC] (ML7) films

To illustrate the effects of process dynamics on resultant films, two multilayer films were produced using a similar recipe. This similar recipe resulted in two films of different final thicknesses. The recipes are shown in the table 3. The parameters listed for each recipe command line are setpoint execution time, laser energy density and pulse rate, magnetron power, substrate bias voltage, background gas type and pressure. Desired results are also entered in a description field for operator reference. The process recipe shown in the table 3 contains the interlayer as well as multilayer sections shown for two films, Ti-TiC-DLC-20x[Ti-DLC], referred to as ML5, and Ti-TiC-DLC-20x[TiC-DLC] as ML7.

Multilayer Generation Via Process Control

The interlayer film provides a Ti base that is deposited on the substrate prior to multilayer film deposition. The interlayer recipe also includes substrate pre-treatment by Ar etch and outgas heating. In the case of silicon, substrate samples necessary for microscopy, argon etch time is reduced to 15 minutes, as compared to 28 minutes required for 440C steel test samples to avoid substrate damage.

The TiC multilayer recipe shown in table 3 differs for ML7 only in that TiC is deposited by operating the excimer laser at 2 Hz during simultaneous magnetron operation. [13] The Ti multilayer recipe is shown in table 1 for ML5. Simultaneous laser and magnetron sputtering deposition is not performed in the ML5 recipe, so that only Ti interlayer will be deposited.

Recipes were then used to generate multilayer films. Three hours and 5 minutes are required to deposit Ti-TiC-DLC-20x[Ti-DLC] (ML5) and Ti-TiC-DLC-20x[TiC-DLC] (ML7) coatings. Computer timing is to within 1 millisecond, so no appreciable variations in schedule execution occur. During each recipe execution, 35 process parameters are displayed recorded in time so that a complete process log is available for future analysis and reference.

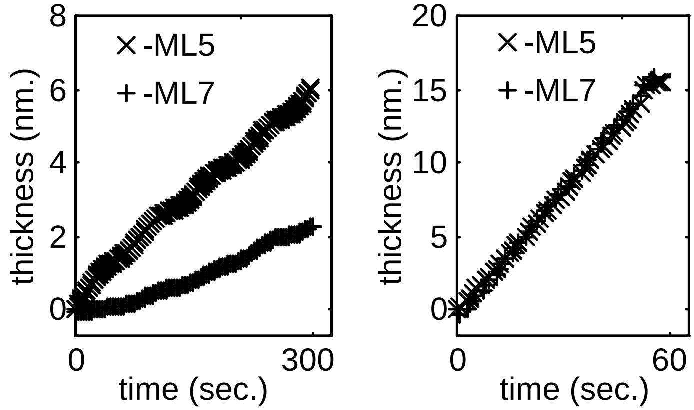

Figure 30 Measured PLD DLC and magnetron interlayer QCM data.

Shown in Figure 30 is actual measured PLD DLC and magnetron interlayer QCM data from Ti-TiC-DLC-20x[Ti-DLC] (ML5) and Ti-TiC-DLC-20x[TiC-DLC] (ML7) recipes. A comparison of ML5 and ML7 quartz crystal microbalance (QCM) thickness and thickness rate data is shown above, for both magnetron and PLD diamond-like carbon deposition. The recipe steps are identical for diamond-like carbon, yet a reduction in deposition rate is noted for ML7 as compared to ML5. Direct comparison shows approximately a factor of 1.8 between deposition final values. Measured PLD DLC rates of 0.01 to 0.05 nm/sec for ML5, and 0.001 to 0.01 nm/sec for ML7 were calculated. A sinusoidal pattern is also present due to a combination of QCM off-axis location and target rastering effects. Measured QCM deposition rates are much less than actual deposition rates at the sample location. This measurement error is due to the characteristic PLD DLC directional plume combined with QCM off-axis location.

Likewise, QCM data collected during the magnetron step in the multilayer recipe shows no appreciable reduction of deposition rate for either ML5 or ML7, even though ML7 includes PLD operation, while ML5 does not. QCM data is overlapping, indicating no appreciable difference in magnetron deposition rate for either run. A measured Ti magnetron deposition rate of 0.3 nm/sec for both runs is an order of magnitude greater than PLD DLC off-axis measurements. No periodic variations were found in the magnetron QCM data.

A DLC deposition rate reduction is apparent in the ML7 film, resulting in less thickness for each PLD diamond-like carbon layer. No appreciable change is present in the magnetron sputtering recipe step execution for either ML5 or ML7. Observed 324 nm LIF spectral emission during the PLD phase of recipe generation also show a reduction in spectral line emission and a subsequent reduction in carbon deposition in ML7 as compared to ML5.

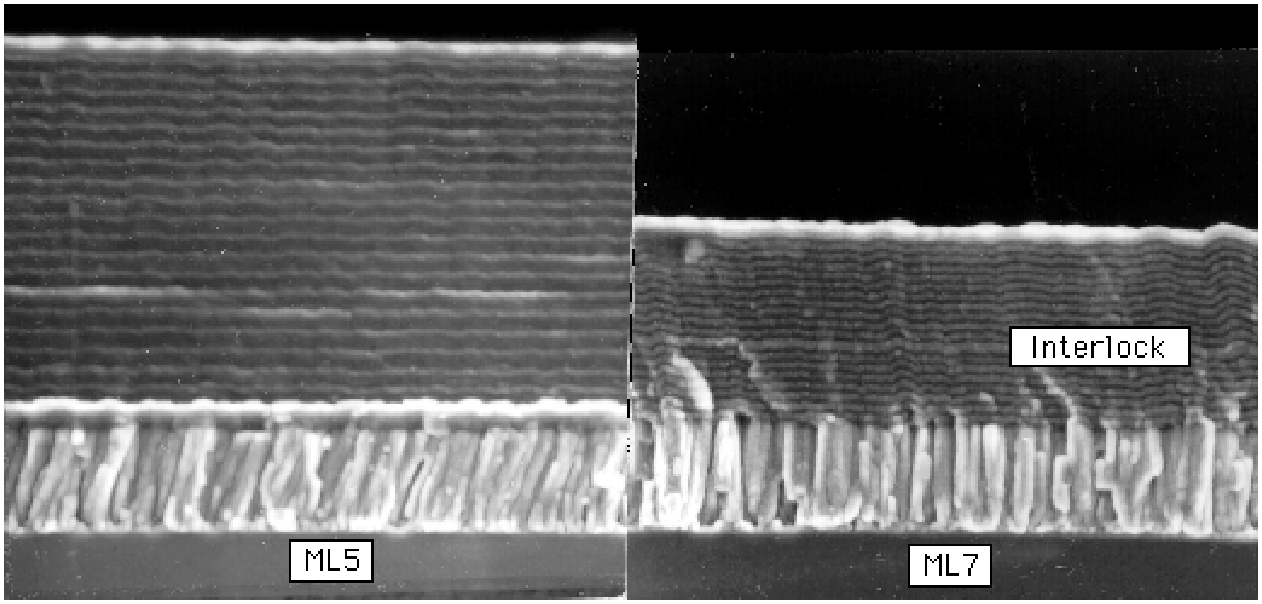

Figure 31 Scanning electron micrograph comparison of Ti-TiC-DLC-20x[Ti-DLC] (ML5) and Ti-TiC-DLC-20x[TiC-DLC] (ML7)

Shown in Figure 31 are edge wise scanning electron micrograph comparison of Ti-TiC-DLC-20x[Ti-DLC] (ML5) and Ti-TiC-DLC-20x[TiC-DLC] (ML7) recipe generated gradient multilayer films showing thickness disparity due to laser deposition window coating. Also indicated is laser interlock. Similarly, representative LIF waveforms are digitized during PLD DLC in vacuum, and PLD DLC with an argon back pressure [16]. A reduction in ion velocity is indicated with an increase in Ar backpressure. Less spectral line, intensity was also apparent in ML7 as compared to ML5, indicating a reduction in PLD DLC laser ablation. Integrated PIN diode laser energy density data shows no reduction in chamber laser entrance energy density for ML7 PLD DLC recipe, indicating laser entrance window coating, and reduction of carbon deposition.

A comparison of two films deposited show that for a reduced thickness value and reduced spectroscopic intensity, a marked reduction in final multilayer thickness is apparent. Edge SEM images of deposition on silicon wafer for two films are shown above.

Based on QCM and LIF data combined with laser energy data, the change in final thickness is due to window coating and subsequent decrease in PLD deposition rate. Close examination of the previous scanning micrographs indicates a missing or reduced DLC multilayer at number 8 from the interlayer in ML7. Post-film deposition examination of process parameters indicates a laser heat interlock occurred, causing a reduction of PLD DLC deposition for this multilayer. The interlock process data is shown in the following Figure 31.

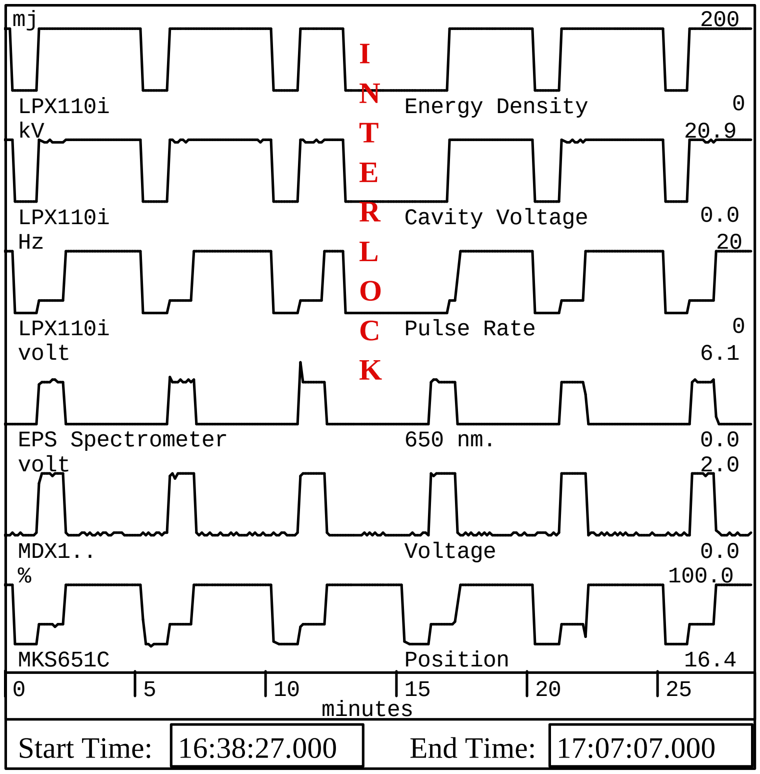

Figure 32 Process parameter fault, showing laser thermal interlock and shutdown.

Shown in Figure 32 is a process parameter fault, indicating laser thermal interlock and shutdown, causing partial loss of diamond-like multilayer film numbers eight in Ti-TiC-DLC-20x[TiC-DLC] (ML7) film. This film was analyzed and rejected due to this system fault, but served as an example of how data collection and archiving can pinpoint system failures, determining either a catastrophic failure, or harmless fault.

PLD Process Control Conclusions

The process control system presented here is capable of producing metal-carbide-DLC gradient materials, and is capable of incorporating them into multilayer coatings, consisting of multilayer stacks of nanolayer composites. Empirical process models in the form of recipes are found that manipulate the PLD process to generate multilayer films of desired composition and structure creating a potential for producing a new class of materials.

Recipe process control and automation make combinations of several complex thin film processes tractable. This, combined with characteristic high ion species velocities previously measured for DLC PLD (100 nm/psec) permit direct atomic combination of PLD DLC with slower deposition processes. Investigation of new material gradients and multilayer heterostructures [36] become possible that may not otherwise be feasible.

Section 4d. Preliminary tests using gas control of a process

Control and Sensor Problem Statement

Preliminary tests were conducted using gas control of a titanium nitride cathodic arc process for incorporation into the MSPLD process. With the existing instrumentation available, it becomes possible to incorporate gas regulation in MSPLD deposition. The MSPLD system must have gas regulation such that transients in gas pressures can be reduced, since the magnetrons are operated in a background gas such as argon or nitrogen, but the PLD deposition is operated in an ultrahigh vacuum environment. The process control must be capable of changing the chamber pressure from 1-10 mT to 10-8 Torr for each layer. In addition, transition pressure must be regulated and a pressure gradual transition must be operated over time with each multilayer growth. In addition, the process must be controlled for the initial boundary interlayer to transition the substrate to the film. Therefore, a control must be available to operate the magnetron deposition simultaneously with the PLD deposition. While gas control of superconductor deposition processes were another example of gas control, some preliminary gas control studies were done using an electric arc deposition process. This information is included to show what sort of pressure control is needed for MSPLD.

Electric arc is a thin film deposition process used to deposit titanium nitride thin films on aircraft engine (F-16) and space (shuttle main turbo pump) components. The process was capable of making thin films of high quality over large areas and on large components, but the film consistency varied. In addition, occasionally the arc sources would inexplicably extinguish, causing a defective coating, which results in scrapping the run.

Increases in thin film coating process quality standard requirements, such as national aerospace defense contractors accreditation program (NADCAP) and ISO 9000 have forced improvements in existing deposition process technology as well as process run information. Increases in process data mangement, thus indicating performance are required to provide customer information as well as diagnostic capabilities.

In situ process control of cathodic arc provides a method of consistent film deposition by unmasking internal process dynamics that are otherwise not observed by sensors at the process boundaries. Increased film quality and yield have been demonstrated using emission fluorescence spectroscopy for *in situ* feedback control. In the present studies, feedback control has demonstrated stabilized process performance superior to that of pressure regulation, enabling the compositional changes required for multilayer films.

Further improvement in cathodic arc deposition systems requires sensors that indicate more closely, what is occurring during a film deposition. Even this information is not useful if it is not applied directly to the process in some way to improve the deposited material. The cathodic arc deposition process is a popular method of depositing thin films (1-10 mm.) of tribological materials for a wide range of applications, ranging form space to automotive needs. Although processes that are capable of depositing films of good quality are commercially available, current processes unfortunately rely on boundary conditions such as pressure regulation, arc currents, and voltages to stabilize the coating composition, while still leaving internal process parameters such as plasma condition uncontrolled. For deposition of multilayer films, additional *in situ* sensors must be combined with a process control recipe in order to determine and stabilize the stoichiometric growth in real time.

Feedback control of *in situ* sensor data is one method of stabilizing a process internally. [37] The objective of this study is to identify an *in situ* parameter that would be suitable for regulation and control of material quality, and then design a system to verify *in situ* feedback of this chosen parameter. Using this method, it is possible to reliably deposit multilayer films with varying composition by adjusting control setpoints in real time.

Process Instrumentation Experimental Approach

A number of parameters of the cathodic arc affect the final film outcome and morphology. These include arc currents and voltages, deposition gas pressure, substrate temperature, substrate bias voltage, and substrate preparation. During operation of the arc, a plasma is generated, and is sustained by a gas pressure. The particular gas type affects the final film composition. In the case of titanium nitride deposition, nitrogen gas is used to sustain the arc. Spectroscopic emission from this plasma can be used as an *in situ* analytic method of determining film deposition characteristics. A system which uses arc emission to vary inlet gas flow is depicted in Figure 33.

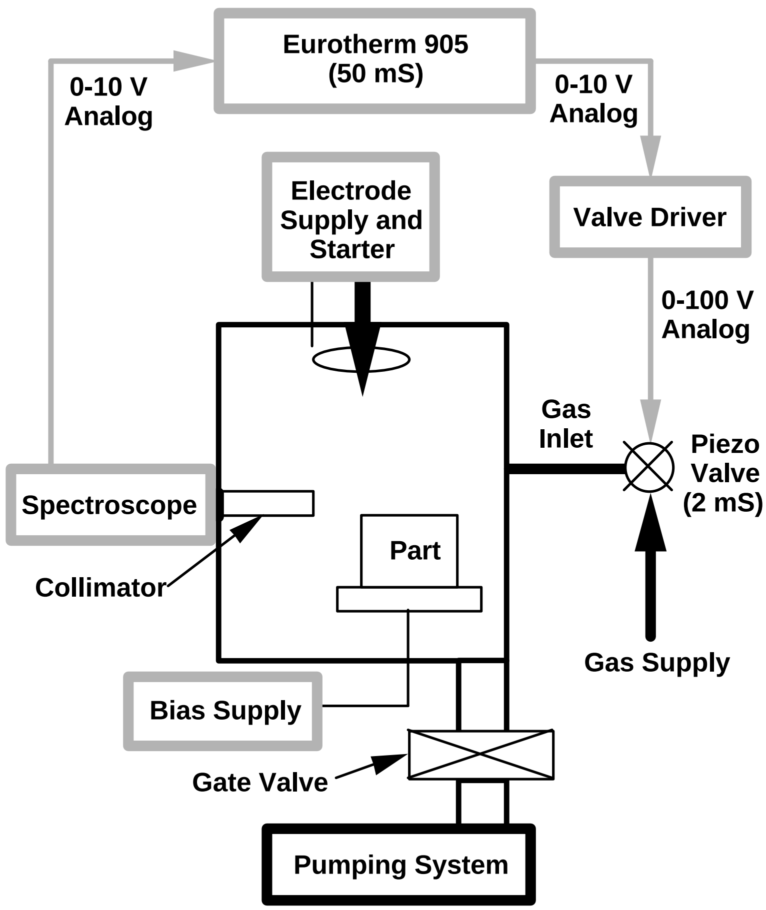

Figure 33 Cathodic arc process control using in situ process control by plasma emission spectroscopy.

An optical emission spectroscope is used to monitor the Ti* (Ti I) ion plasma emission at 650 nm wavelength. Other spectral lines were tried, but this line had sufficient intensity to be detected reliably, and was unique. Utilizing this spectral line intensity to actuate a high-speed piezoelectric valve to change chamber partial pressure was found to directly sense deposited film composition. In order to determine spectral line control, the arc current and voltage are regulated in these experiments, while gas, pressure is varied. Films produced by *in situ* closed loop control were then analyzed by *exsitu* tests to determine variations in stoichiometry, as well as actual tribological properties.

Estimated gas residency times were calculated from pumping rates and mass flows. From these calculations, PID tuning was performed using a Ziegler-Nichols method [38] at setpoint maximum and minimum. The tuning parameters were then modified based on process performance. Acceptable values of proportional band and integral reset used were 600% and 15 sec. respectively. These values proved adequate to stabilize the process involving processing intervals of 4 hours.

Unlike spectral emission regulation used in magnetron deposition or CVD, the spectroscopic signal from the cathodic arc process is noisy, and is of varying probability density, due to the erratic behavior of the arc on the electrode. Furthermore, the spectroscope probability density is a function of gas pressure, with less noise apparent at lower pressures. Spectroscopic intensity is also affected by cathode temperature and amount of cathode poisoning. Thus, pre-filtering of the spectroscopic signal is necessary for stable control performance.

A preliminary solution is to use a 3-pole Butterworth [39] low pass filter, combining filter properties of having the least amplitude and phase distortion for a given stop band attenuation. A more sophisticated method involving arc plasma intensity signature identification could be used. A mapping, perhaps a functional-link [18] neural net, could be used to identify plasma species intensity and correlate the emission to deposited film characteristics. The additional functionality would require an increased effort, and was not supported.

Using the preliminary design, the piezo-valve mechanical response time is limited to 2 ms, so the maximum actuator bandwidth is limited to 500 Hz. Spectroscopic sensor temporal data analysis showed a limited information response time of approximately 100 ms, indicating that a cutoff frequency of 10 Hz was required for this particular chamber and pumping system. The estimated power-spectral density taken over several process runs showed that a process integration time of 25 seconds is sufficient to remove variation due to arc motion. Thus, a slower response time valve could be used.

Stabilization of the process arc was ineffective when variation reductions with a time interval of less than 100 ms were attempted, indicating estimated information bandwidths to be accurate. An increase in compensator gain only destabilized the arc and generated excessive piezo-valve actuation. A reduction of the spectroscopic pre-filter cutoff frequency below 10 Hz reduced the overall controller response.

Two process run data sets collected with ‘in-house’ developed data acquisition software, referred to as InfoScribe, coupled to Labview are shown in the following graphic, showing closed loop control of the electric arc in comparison with open loop arc spectral line intensity. Spectroscopic data combined with actuator effort are shown in comparison with pressure regulation for intensity setpoints of 10% (b) and 90% (d) respectively. As can be seen in both b and d, pressure regulation indicates a variation of up to 20 % for spectroscopic line intensity occurs due to random arc motion. Because of *in situ* process control, spectroscopic line intensity shows less than 5% variation across several runs.

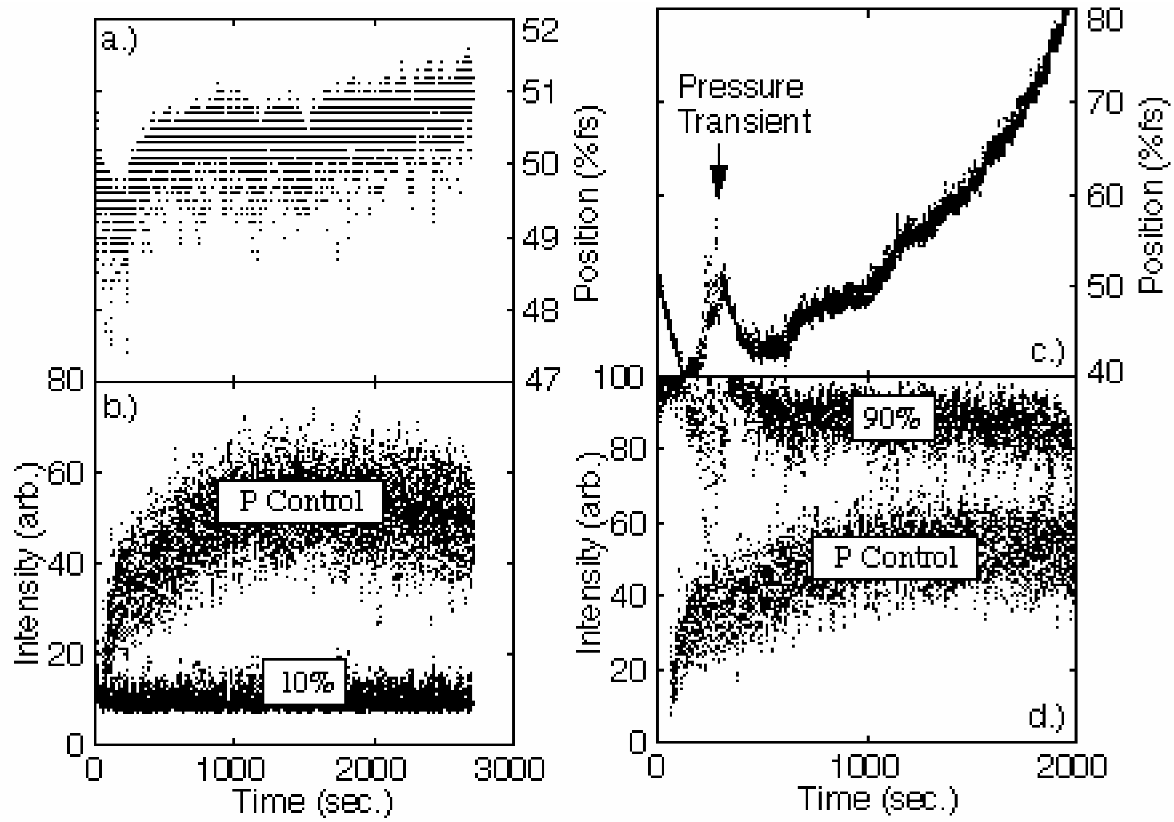

Figure 34 Comparison of spectroscopic signals from in situ process control and pressure control.

Shown in Figure 34 is a comparison of spectroscopic signals from in situ process control and pressure control comparing gas regulation and spectroscopic regulation of the cathodic arc process. These were as follows:

a.) actuator position at 10% intensity setpoint,
b.) in situ control performance at 10% intensity setpoint,
c.) actuator position at 90% intensity setpoint,
d.) in situ control performance at 90% intensity setpoint.

Actuator effort for both runs is also shown for setpoint regulation of 10% (a) and 90% (c) respectively. Typical actuator effort starts at a low value and steadily increases during the run, indicating a higher pressure is necessary to maintain spectral line intensity. A pressure transient, shown in c, was later found to be due to an instability in the nitrogen pressure gas manifold that feeds several production chambers. This transient was found to occur whenever another deposition process that shared the same manifold was initialized. This problem has since been corrected, but was unknown prior to installation of the *in situ* process control sensor.

Control and Instrumentation Results and Discussion

Once the *in situ* spectral line process control system was performing correctly, experiments were performed to compare films produced by *in situ* process control with films produced by pressure regulation. Pressure regulation used standard values and practices developed by Hohman. Spectral line emission controller setpoint boundaries were defined as 0% intensity equating to the nitrogen gas pressure that extinguishes the arc, and 100% intensity as the nitrogen gas pressure that causes electrode poisoning. Calibration between each run was also checked by utilizing a calibrated white light of fixed intensity that is placed within the dark, sealed chamber. The spectroscope was then checked for drifts and inaccuracies prior to each process run. Samples using both pressure regulation and *in situ* process control were produced to compare the coatings:

1. Coatings were produced using a standard TiN deposition procedure used by Hohman with mass-flow and Baratron gage pressure regulation. This method of pressure control uses mass flow control in a proportional plus integral scheme. Spectral line intensity was also recorded during these coating runs. This method was used to produced samples designated as "TiN reg".

2. Coatings were produced using a standard TiN deposition procedure used by Hohman, but with control of gas using closed-loop *in situ* plasma emission process control feedback instead of gas pressure regulation. In these runs, nitrogen gas pressure is allowed to vary while spectral line intensity is regulated. Titanium spectroscopic line intensity is then used to generate coatings at three setpoints, with two process runs performed at each setpoint. The spectral line intensity samples are produced as follows:

Coatings produced at 10% relative intensity of control signal, designated as "TiN 10%".
Coatings produced at 50% relative intensity of control signal, designated as "TiN 50%".
Coatings produced at 90% relative intensity of control signal, designated as "TiN 90%".

In total, four different TiN coatings were prepared in 8 runs, to provide duplicate samples of each type. Several test runs were also performed prior to these sample comparison experiments to verify proper controller tuning and performance for both pressure regulation and *in situ* plasma emission process control. Coating chemical composition was then evaluated using an X-Ray photoelectron spectroscopy (XPS) with an M-Probe instrument. Data were recorded with 0.2 eV resolution. Samples were sputtered with a 1 keV Ar gun for 10 min. to remove any surface contamination.

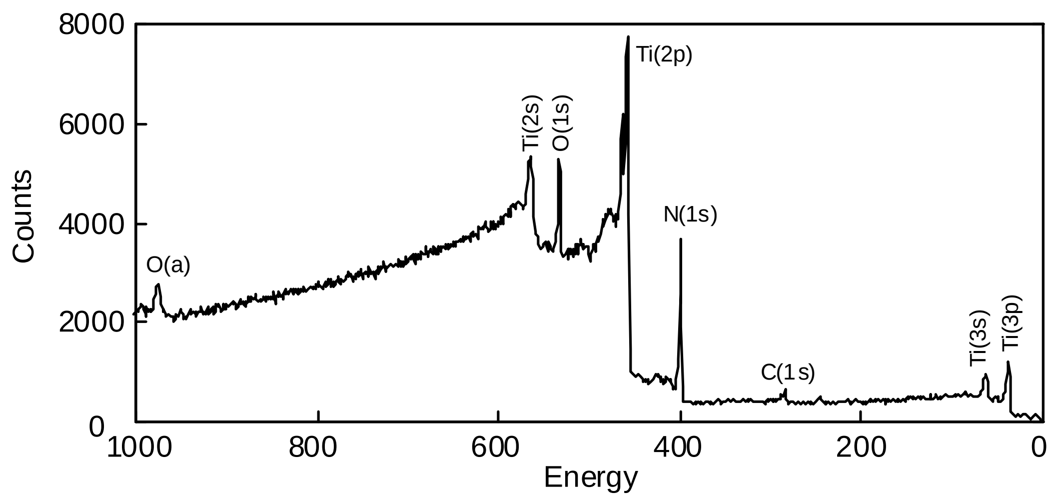

Figure 35 Typical XPS spectrum of film produced by in situ process control.

Typical spectrum of the coating elemental bonding energies is shown in Figure 35. The spectrum shows the presence of Ti, N and O with traces of Ar and C. The indicated argon presence is due to the XPS surface etch procedure. Traces of carbon are typical for deposition systems with diffusion pumps. Possible origins of the indicated oxygen contamination are due to oxidation of nonreacted Ti in the coatings with oxygen from the atmosphere. Another possible source of film oxygen is from residual water pressure in the chamber. Residual water is more likely, given the large physical size of the deposition chamber used.

Sample ID	Ti at.%	N at.%	O at.%	Ti/N ratio
TiN reg	49.10	38.73	12.80	1.27
TiN 10%	51.60	40.85	7.55	1.26
TiN 50%	48.19	42.65	9.16	1.13
TiN 90%	46.50	43.54	9.50	1.06

Table 4 Composition analysis of films produced under pressure control ("TiN reg") and in situ process control (TiN 10%, TiN 50%, TiN 90%)

A summary of film chemical composition for the main elements Ti, N and O of several films is shown for comparison in the table 4. A ratio of Ti/N was also calculated and is included. It can be seen that a coating produced by pressure regulation is slightly over stoichiometric in respect to titanium. However, the excess of nonreacted Ti is small (no metallic Ti was registered with XPS). Possibly some of the excess Ti atoms are dissolved into the TiN phase, while others formed oxides, present after each deposition, which typically precipitates out as a powder.

Coatings deposited with plasma emission control at 10% and 50% signals were found to be slightly over-stoichiometric as well. Coatings produced with 90% signal were found to be close to stoichiometric in chemical composition. It was also noted that less precipitated powder was present after each deposition run, and less oxygen contamination was present for coatings produced with plasma emission control.

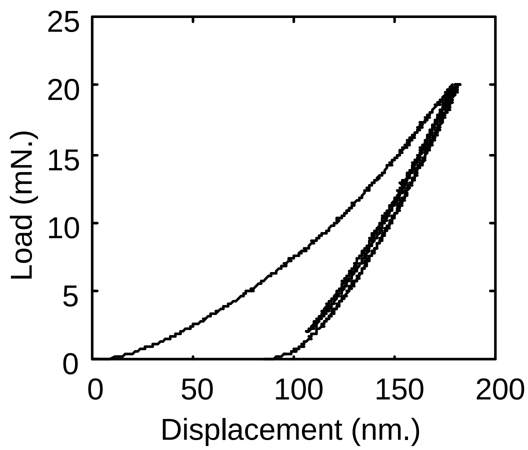

Figure 36 Typical nano-indentation curve measured on films produced by in situ process control and pressure control.

Hardness and elastic modulus of the produced TiN coating were estimated. For this purpose, a Nanoindentor II instrument with a Berkovich indentor was used. In these tests, an indentor was loaded with a force of 20 mN and load-displacement curves were recorded. Typical procedure is shown in Figure 36. The curve is generated by increasing load to the maximum, unloading to 10% load, 50 sec holding for a thermal drift correction, loading to maximum load again and final unloading. The hardness data were taken for maximum load at a penetration depth of approximately 180-200 nm. Young's modulus is then estimated from the final unloading curve slope.

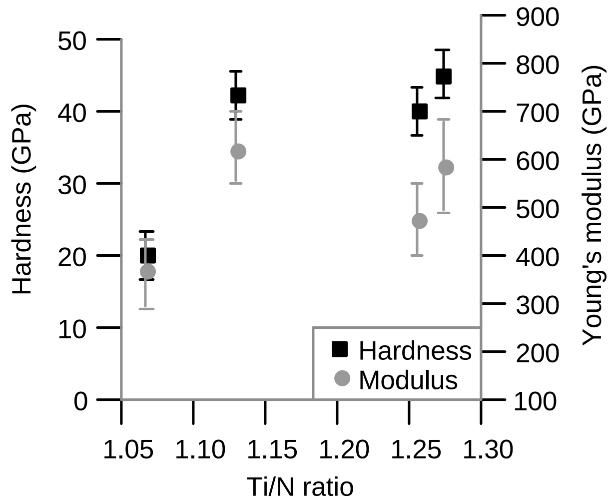

Figure 37 Mechanical test results of films produced by in situ process control and pressure control.

Results of the films produced are accumulated in the previous figure for hardness and elastic modulus as a function of Ti/N ratio. These data suggest that all coatings with slightly over stoichiometric Ti content have approximately the same hardness of around 40 GPa and a modulus of around 550 GPa. This includes TiN produced under pressure control, and TiN produced under spectroscopic regulation of 10% and 50% intensity, as indicated at the top of Figure 37.

The stoichiometric TiN hardness produced under 90% spectroscopic regulation is reduced to about 25 GPa, which is a typical value for TiN deposited by cathodic arc. [40] These results suggest that a slightly over-stoichiometric composition is favorable for achieving optimum mechanical properties. This agrees with results of previous investigations [41],[42], which show that for TiN coatings with insufficient N content, the structure is denser, and consists of fine grains and a developed dislocation network. Coatings of this composition are also known to have higher levels of compressive stresses, which also affect measured hardness values.

Cathodic Arc Process Control Experimental Conclusions

Consistent coating that can be produced over long times is a practical advantage of *in situ* spectroscopic intensity process control. *In situ* sensing increases process stability by sensing internal process parameters where the deposition is actually occurring, and not at boundary conditions, such as pressure. These issues are important when coating large parts of high cost. In some cases, the process is required to generate a uniform coating over a part of complex shape and large size, as well as high cost. Risking stabilization of a deposition process by pressure alone opens up the chance of scrapping the part, and ultimately eliminating the customer base.

In situ process control of cathodic arc allows for more consistent film deposition by unmasking internal process dynamics that are otherwise not observed by sensors at the process boundaries. Consequent film quality and yields are increased, as well as increased process attribute records and run data. This additional process information increases the operator's knowledge of overall process condition as well as providing additional data to satisfy new standards, such as national aerospace defense contractors accreditation program (NADCAP) and ISO 9000.

In situ spectroscopic intensity process control provides an additional advantage by better informing the operator of the true process condition, as well as attempting to compensate for any unforeseen variability not apparent at the process boundaries. Comparison of films show a reduction of oxygen in all cases of films produced under *in situ* process control as evidenced by a reduction in powder generation, while maintaining desired mechanical properties. When high process repeatability is demanded over long time periods across several runs, these advantages demonstrate *in situ* spectroscopic intensity process control is superior to pressure regulation on Cathodic arc production systems.

Section 5. Installed hardware system description

The MSPLD system, as delivered, is capable of simultaneous PLD and magnetron sputtering under automated control. This unique system is capable of several advanced functions that are not possible on any other machine in the world, and currently utilizes the largest deposition area for a PLD machine, 4 inches. Operation of each subsystem was verified as well as the operation demonstrated to the customer, including deposition of both PLD and magnetron modes of operation and all other manner of system functionality. What follows is a brief description of the system's features and what makes this system unique as it was installed at the customer's facility.

In order to accommodate production levels of film generation, this machine has a large cavity size, approaching a cubic yard of UHV volume, which is easily accessible via a single hinged front door. This enables the entire contents of the machine to be accessed by simply opening the door, making for quick service and reloading of coating substrates. An open view of the chamber cavity is shown in Figure 38, with the rod coating fixture installed.

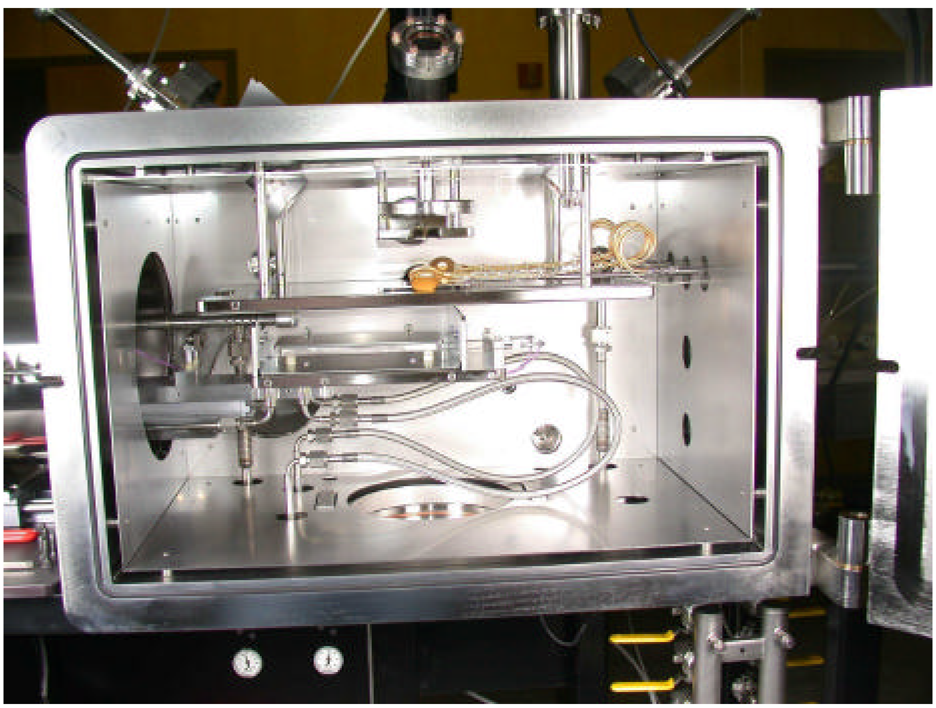

Figure 38 Chamber Cavity

The entire chamber cavity can be pumped down at ultrahigh vacuum or to background sputtering pressures under automated computer control. The large 4-inch area can be deposited with both the PLD and dual magnetron sputtering modes of operation occurring simultaneously. Dual 3" magnetrons can deposit uniform materials at mTorr background gasses, and then the entire system can automatically be changed to operate at UHV for PLD deposition of high SP3 bond carbon. Pressure and mass flow of background gasses are both PI feedback controlled to setpoint regulate both mass flow of gas species as well as chamber pressures during dual magnetron operation. All subsystems are computer controllable and tied to the computer interface infrastructure for recipe and automatic multilayer generation.

Multilayer MSPLD occurs while the system substrate heater is operating. Tests were done at the installed site, heating the components up to 500 degrees centigrade, with higher temperatures possible at reduced component life. The heater design is totally contained to reduce radiant heat by several heat shields and water cooling jackets to keep only the parts to be coated at high temperatures. The heater is PID temperature controlled by a Eurotherm PID temperature controller, operating the heater plate to give temperature regulation down to a single degree. This heater is shown in Figure 39, below the rod mounting fixture.

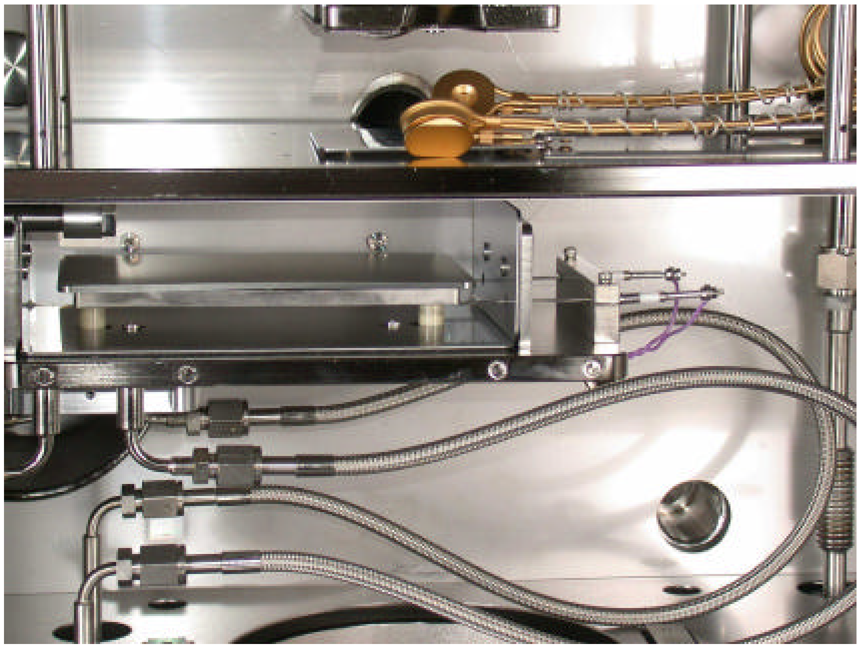

Figure 39 Rod Mounting Fixture

Also visible are the gold plated dual point quartz crystal microbalances capable of angstrom resolution across the deposition field, providing two point spatial resolution. These microbalances are also water cooled and integrated with computer control infrastructure of the MSPLD machine. Deposition processing can be monitored using these microbalances, so that both the PLD and magnetron sputtering deposition rates and characteristics can be observed during multilayer generation.

This system has two easily removable substrate coating stages designed to coat 1/2” ball bearings by the rolling (slosh) method, up to 3 -1/2” diameter rods of up to 6” length, and bearing races of up to 3 1/2” diameter. These stages are removed via a rail system and can be heated to above 500 degrees Celsius, as well as up to 1000 volts of DC bias applied as they rotate and are moved back and forth by computer control to generate a uniform coating. The substrate bias voltage control is tied into the computer interface infrastructure of the MSPLD system so that computer control can effect bias and rotation to generate uniform coatings. The bias connections are shown in Figure 40 for the rod stage, showing the UHV stepper motor and bias interconnections as well as cooling lines for the UHV rated stepper motor.

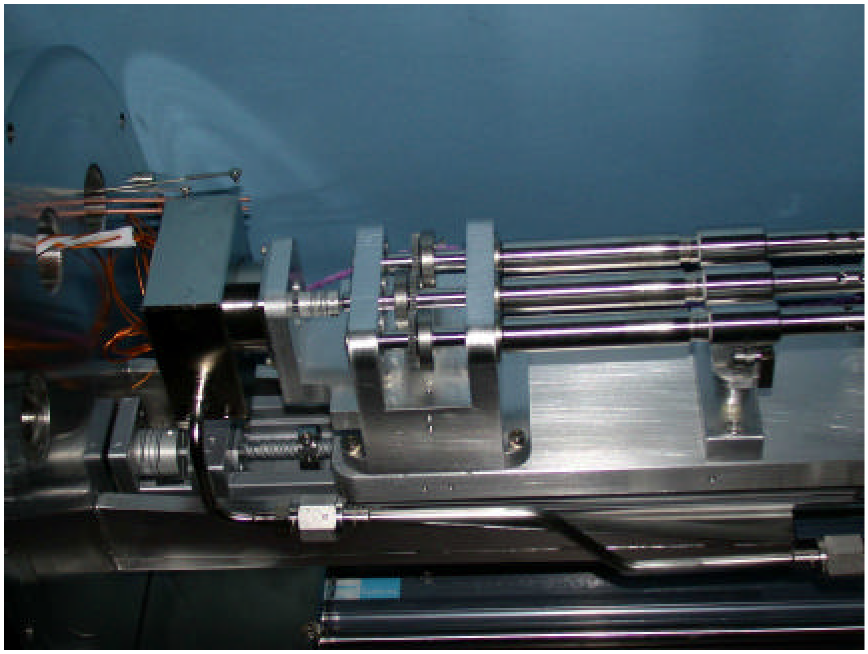

Figure 40 Rod Stage UHV Stepper Motor

A second stepper motors controls the stage movement for even coating of the rods. These motors are operated by a PLC industrial grade controller which also the intelligent window motor. The PLC motion controller is also tied into the computer interface infrastructure of the MSPLD system as well as having it's own user interface. Computer code was written to demonstrate operation of both stages to the customer's satisfaction, as well as providing a development environment for the customer to write their own code. These were all tested and functionally demonstrated to the customer.

The two stages are easily removed via a rail system to accommodate coating rods or bearings and races. Shown in Figure 41 is the rod stage being inserted into the chamber via the chamber's rail system, preventing excessive handling of the stages.

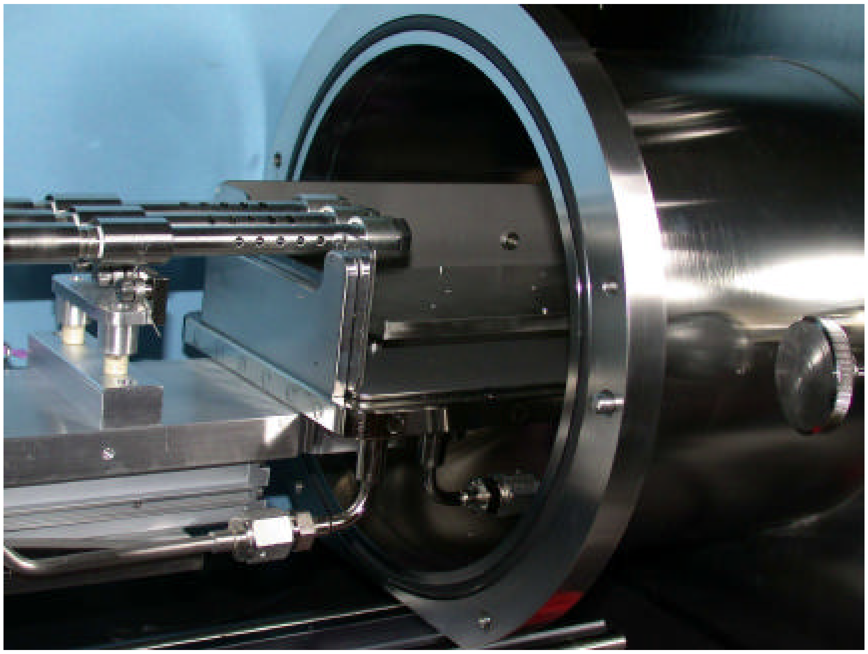

Figure 41 Stage Port

The MSPLD system also has two systems to enable long operating times for the PLD system optical train. The optical train utilizes intelligent window functionality with integral power monitoring capabilities as well as a high-speed galvanic laser spot rastering mirror for PLD operation. Operation of these subsystems was demonstrated to the customer and is as follows.

The intelligent window prevents laser beam entrance window degradation from window coating. A pneumatic energy coupler is computer controllable so that during operation, the laser power can be monitored inside the vacuum system. If the laser power entering the chamber is found to be inadequate due to window coating, the input laser UV window shield can be rotated to remove the coated portion from the laser light path. This operation is called the intelligent window, and is computer controllable and tied to the MSPLD computer control infrastructure to permit laser energy at the target to not degrade over time with window coating.

Also, a high-speed galvanic mirror randomizes the laser spot placement on the target. This is done so that target heating is minimized, preserving the target morphology and structure. This has been found to reduce variance in the ablation threshold for the PLD laser deposition. The PLD optical train with intelligent window and galvanic raster mirror subsystems are shown in Figure 42.

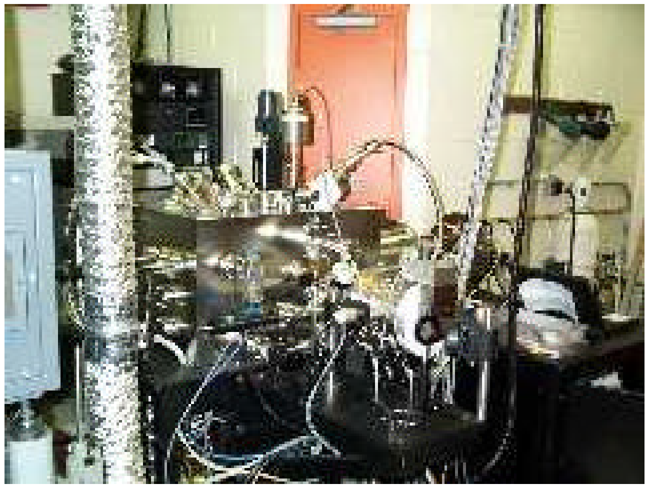

Figure 42 Raster Mirror

The MSPLD system also utilizes atomic emission spectroscopy for in-situ plume diagnostics. A dual channel spectroscope and high speed digitization oscilloscope is used to capture plume emissions and species velocities and densities. A custom dual channel spectroscope was constructed and demonstrated. This is shown in Figure 43, and is installed on the door of the chamber.

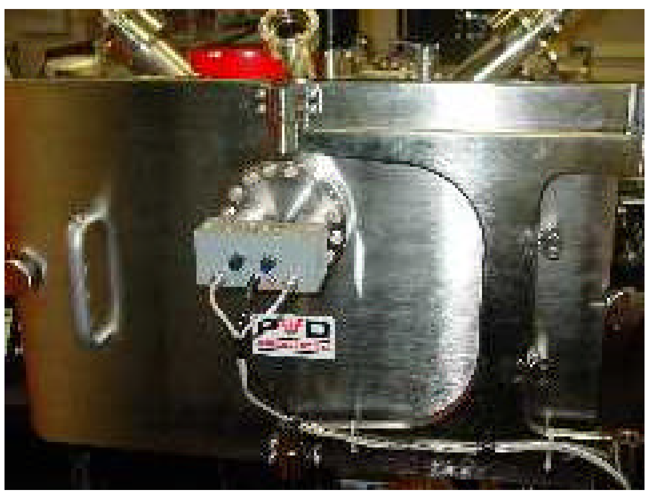

Figure 43 Atomic Emission Spectroscopy

This custom spectroscope utilizes dual internal adjustable 1 kV DC bias supplies as well as end view high gain PMT's used to sense weak emission species. The unit was installed and tested as well as several filters for carbon species were delivered. This was connected to the high speed Hewlett Packard Infinium digitization oscilloscope that was also tied into the MSPLD communication infrastructure.

The MSPLD system instrumentation is on par with most research chambers utilized at the customer. Here is a short description of the equipment as installed in the MSPLD main control rack, as tested and demonstrated. This rack contains the following controllers, from top to bottom:

(rack top)
• Rack air circulation fans.
• Eurotherm 905 PID substrate controller and driver remote control.
• Granville Phillips 307 UHV ion gage controller and dual convectron gages.
• Dual XTC quartz crystal microbalance controllers.
•VAT pressure controller, with baratron gage and stepper motor actuated UHV gate valve.
• Turbomolecular pump controller.
• Galvanic mirror raster control panel.
• Emergency stop and safety interlock panel.
•HP Infinium spectroscope digitization oscilloscope.
• Bottom panel containing power supplies, and logic, bus, etc.
(rack bottom)

The main control rack is shown Figure 44, as installed and in operation at the customer's facility:

Figure 44 Main Control Rack

All these instruments and subsystems are integrated onto the computer control interface infrastructure, and are computer interface capable. These are matched to many of the existing code in LabVIEW, providing for in-house code development and maintenance.

Also can be seen to the left of the tall rack is the process control data server short rack. This rack contains the Marathon RISC computer, keyboard drawer and monitor. It runs the InfoScribe v.2. data server software, accessible via LabView subroutine calls and TCP/IP http web interface on a dedicated MSPLD system network. This was originally designed to be tied to the customer's in-house network to provide area wide MSPLD process data access via web browser interface.

The chamber stand also contains a half-rack of equipment for operation of the MSPLD system. This half rack contains the following instruments shown in Figure 45:

- Emergency stop panel.
- Pump down, interlock, and chilled water status indicator panel.
- MKS mass flow controller and hot wire mass flow actuators.
- Control logic enclosure.
- Substrate heater driver control panel.
- Magnetron 1, Magnetron 2, and Substrate bias power supply panels.

Figure 45 Chamber Control Rack

The chamber control rack also contains the PLC motion controller that operates the stepper motors for the stage motion as well as the intelligent window function. The back side of the chamber control rack contains the system interlocks on water, system vacuum, and HV, as well as additional control logic and power distribution and fusing. This is shown in Figure 46.

Figure 46 Power Distribution Rack

This back panel also contains power supplies and some relay control and DIN rail components for proper operation of the MSPLD system.

The overall system was fitted into the lab as designed. The system cleared all doorways and elevators, and was installed to the customer's specifications, ahead of projected delivery deadlines, so that the steered arc machine could also be installed in the lab. The MSPLD system can be seen, installed, in the following Figure 47, with the steered arc machine barely visible in front of the door.

Figure 47 Installed MSPLD System

As can be seen, there is little room for additional equipment. The data server and monitor short rack can be removed and the InfoScribe server be operated in a "faceless" mode, to remove that rack, and free up some additional floor space. The use of wireways and only a few hanging cable clusters provided for a neat installation of the MSPLD system in limited floor space. Shown in Figure 48 is another view of the MSPLD system in the lab, showing how the integrated optical bench is contained in the chamber stand, and how the optical train is mechanically secure to the chamber. Thus, it only becomes necessary to direct the unfocused laser beam towards the proper spot on the chamber stand. This allows for easy "sharing" of the laser across several processes.

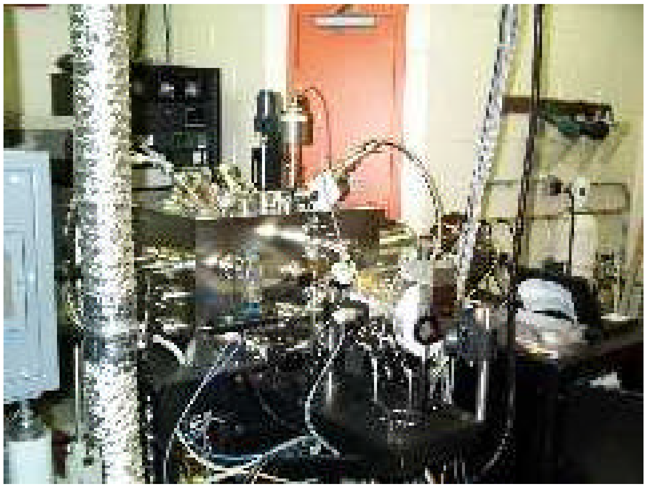

Figure 48 Installed MSPLD System Other View

Figures 49 and 50 were acquired from the process while the magnetron sputtering was active.

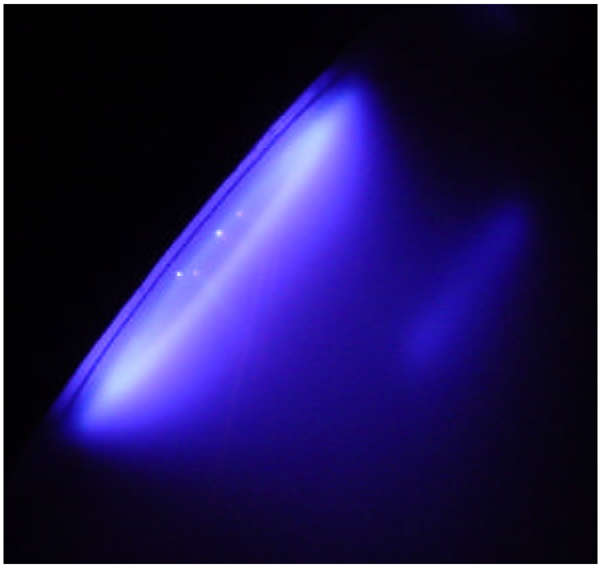

Figure 49 Magnetron Gun

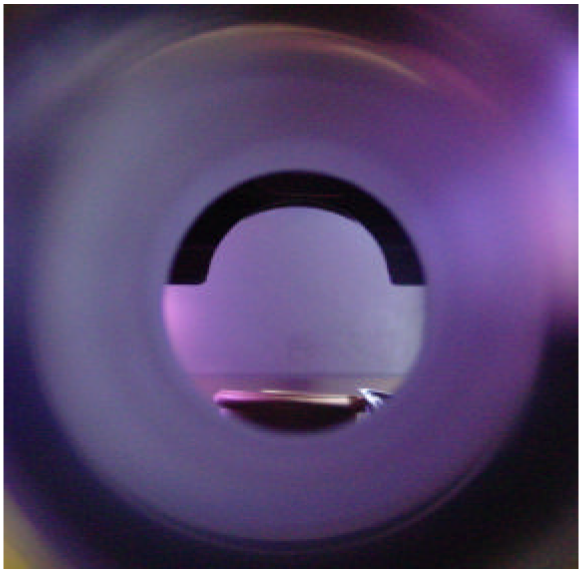

Figure 50 Both magnetron guns

Thus, the MSPLD sytem was installed and demonstrated in it's entirety, to meet all the customer's specifications to their satisfaction.

Section 6. Conclusions

First, any material process does not create or destroy substance. The process merely modifies existing materials into different forms through the use of energy. The characteristics of these new forms of existing material are usually only partially understood through some method of observation of the substance's accidents or physical properties. Furthermore, observation of these physical properties by sensing utilizing some electronic or mechanical device further removes the actual material properties once more from what is seen or surmised based on some indicated value. This is ex-situ observation of the changed substance. The main thrust of this research is to further develop the observation of the material substance change to include the observation of material accidents by electronic sensors during the modification of its substance while it is being changed. The modification is also observed through time. All this sensing and actuation observation is to better understand how a process actually modifies a substance to take a new form.

All processes that are changing, or dynamic, require an understanding of the basic first physical principles that are occurring within the process. Once the first principles of change are understood, at least generally, then the collection of sensor and actuator data brings up ambiguities in what is actually being observed in a particular sensor. The consideration of how any sensor and actuator is coupled into any process requires a careful study and analysis of the basic physical properties that interact with a particular sensor and actuator suite.

Utilization of Computer methods described in this research has permitted a quantum leap forward in materials science by more complete observation of data generated by the sensors. Careful examination of the process sensor data as it responds to actuator change results in a new complete understanding of how the process operates to generate materials. Instead of being restricted to a set of static process parameters, the more complete collection of process data, combined with archiving and categorization of the process sensor and actuator data has and will enable a more complete understanding of how the process actually generates the materials.

Because of the sheer quantity and large size of the data sets from long-term processes such as MSPLD and cathodic arc deposition processes, this information has long been wasted. This research has demonstrated the clear value of process sensor data in the making of new materials and control of new processes that otherwise would be impossible by human means alone. The sheer number and volume creates a dimensional space of process data the overwhelms our ability to visualize the interrelationships of more than three parameters at once. This is only limited by the investment capital required to augment a process with sensors and process control computer and software.

The system has been tested and material has been deposited from both the magnetron and PLD processes. The system has meet the requirements of the contract.

Section 7. Dual Uses

Dual uses for this technology are numerous. Two uses have clearly been demonstrated in the results section of this document to bring about fault detection and improvement in materials in one case and a completely new class of materials in another. The software and hardware developed can be utilized in any place where data is collected, manipulated, and archived in a computer. What can be seen from this research is that information produced by a process sensors and actuators while producing materials can be used to develop the process and utilize it to develop and produce newer materials or product, whatever that may be.

Section 8. References

Lab Task Manager: Dr Samuel J. P. Laube,

Team: Dr A. A. Voevodin, Dr J. S. Zabinski, Dr S. R. LeCair, Dr A. G. Jackson, Dr J. S. Solomon, Dr S. D. Walck, Dr M. S. Donley, Mr A. Korenyi-Both, Dr , M. A. Capano, Mrs E. F. Stark, Mr J. D. Busbee, Dr R.B. Trelease, Students: Mrs C. Strong, Mr K. Keener, Mr J. Masarelli, Mr S. R. Murray

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