Developments in Energetic Processes for Optical Coating Applications

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ABSTRACT

Energetic process development in the production of optical coatings has progressed significantly over the last two decades, permitting the practitioner of thin film coating depositions a wide choice of deposition parameters. Primarily, a series of important advances has occurred in the nearly ubiquitous use of Ion Assisted Deposition (IAD) for the production of high performance optical coatings. Progressing from the rudimentary use of ionized gas technology for pre-cleaning substrates, to the advanced IAD produced telecom filters (DWDM), energetic processes now play a vital role in most optical coating production. The advances in IAD technology culminate in the development of stable and durable thin films for a wide variety of stringent spectral specifications from the UV to the Far IR. The technical progression from IAD use in either sputtered or physical vapor deposition (PVD) processes to the development of Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) technology for low temperature production of thin-film coatings will be discussed.

1. INTRODUCTION

Emerging from the pioneering work during the 1960's of the Soviet Union and NASA in the development of ion thrusters for space propulsion, the Kaufman gridded ion generating source was adaptively designed for thin-film depositions.[1-3] Early work with a modified gridded ion source proved that lower voltages and higher beam currents were most suitable for Ion Assisted Deposition (IAD) applications.[4]

Therefore, an end-Hall ion source, described by Kaufman *et al*, was built for Optical Coating Laboratory Inc. (OCLI).[5] The author had the privilege of being the first engineer to put this source into a production application. Specifically, there was a dramatic need to increase the durability of deposited Zinc Sulfide (ZnS) layers. There are two primary crystal states for ZnS, Wurtzite (hexagonal) and Zinc Blende (cubic). In conventional Physical Vapor Deposition (PVD) substrate temperature is the first order effect for determining the durability of the ZnS layer. With the application of this first end-Hall ion source a ZnS cubic close packing (Zinc Blende) thin-film microstructure was attained. Astonishingly, the revolutionary benefit of this process was that the obtained film performance appeared almost independent of the substrate temperature. This first end-Hall IAD film was an order of magnitude more durable than any conventionally deposited ZnS film produced at OCLI up to that time.

Practically speaking, the end-Hall source has two crucial attributes that separate it from the typical gridded ion sources. The broad ion beam (30^{o} half-angle) produces substantially uniform ion density over a large substrate plane. Gridded ion sources, on the other hand, produce a collimated beam that can be broadened only slightly. In addition, gridless ion sources operate using lower voltages (50 to 300 eV) and much higher currents (up to 13 Amps) than even a modified gridded source. Today, there are a number of end-Hall ion sources available for IAD applications. However, each source has unique and common features that need to be evaluated for suitability by the process developer for their specified applications.

Summarizing generally, it has been demonstrated that bombardment of a growing film with energetic ions enhances the performance of the thin-film properties for optical filter applications. Improved film adhesion is achieved by ionic bombardment of the substrate prior to film deposition. Densification of the film, deposited on either heated or unheated substrates, is achieved with IAD. Other film properties may be positively influenced by this technique, such as: residual stress modification; surface morphology modification (crystal orientation, smoothing, and grain size); enhanced optical performance (stable refractive indices and low-absorption); and durability.[6-8]

In parallel to the early work with IAD, other technologies were developed to produce high performance thin-film coatings for a variety of applications and substrate materials. In addition, advanced monitoring and process control systems were developed during the Telecom filter boom that are now available for use in new deposition process regimes. Particularly, technologies like Ion Plating; Jet Vapor Deposition; Plasma Enhanced Chemical Vapor Deposition (PECVD); Post-IAD reactive sputtering technology; Rotating Cylindrical Magnetron Sputtering; Biased Target Ion Beam Deposition; and Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) are all emerging for niche applications in the thin-film deposition world.

2. ENERGETIC TECHNOLOGIES

2.1 Ion Plating

Even though Ion Plating was introduced about four decades ago it had limited applicability in the precision optical coating arena due the non-uniform deposition character that is induced by the ion beam impinging on evaporation material. Instead of a normal cosine distribution from the evaporation crucible the plume is distorted into a parabolic topology that is problematic to resolve by conventional substrate masking. Recent work by A. Andres suggest that Ion Plating can be combined with Cathodic Arc Deposition into a hybrid process that may have advantages for advanced deposition applications.[9]

2.2 Jet Vapor Deposition (JVD)

The novel technology was introduced in the early 1990's, but has not been widely accepted into the thin-film deposition community. In this technology gas is forced past the evaporation source resulting in a jet stream of deposition material growing on the substrate.[10] It may be advantageous to couple the JVD technology with IAD to provide an even higher evaporation rate process for IAD quality thin-films in future applications.

2.3 Plasma Enhanced Chemical Vapor Deposition (PECVD)

Both optical and tribological applications have been addressed by Plasma Enhanced Chemical Vapor Deposition (PECVD) technology. Tribo-mechanical analysis of PECVD coatings has been developed for evaluating the durability of important optical materials like TiO_2 and SiO_2. It was determined by M. A. Raymond *et al* that inhomogeneous (graded index) coatings show improved tribological properties over conventional multi-layer systems.[11]

2.4 Post-IAD reactive sputtering technology

This technology describes a deposition strategy that consist of alternative sputter deposition and oxidation stages in a single system.[12] It has been reported that this technology has undergone continuous improvements. Dual cathode AC sputtering has supplanted single cathode magnetron sources to affect a reduction in the wandering anode problem, in which causes thickness variations along the length of a target. This upgraded system is claimed to be capable of depositing high tolerance complex filters at a lower cost.[13]

2.5 Rotating Cylindrical Magnetron Sputtering

First applied for large area depositions this technology resulted in high rates for titania films. The films described in this application were produced on a 100 inch (2.5m) wide production scale, in-line coater.[14] Precis Design Corporation developed tubular rotating cylindrical magnetron sputtering for the production of high performance telecom filters (DWDM).[15]

2.6 Biased Target Ion Beam Deposition (BTIBD)

BTIBD was developed to overcome some of the process constraints experienced in conventional Ion Beam Sputtering. By applying a negative bias to the evaporation target material, low energy ions from a standard end-Hall ion source are used instead of the high energy ions produced by a conventional gridded ion source. The advantage of the low energy ions resides in the fact that sputtering of the hardware surrounding the target is avoided, resulting in much cleaner deposited films.[16-18]

2.7 Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD)

IFCAD couples Ion-Assisted-Deposition (IAD) with Filtered Cathodic Arc technology to provide a versatile platform for depositing high performance coatings in advancement optical and electro-optical material applicaitons.[19-21] IFCAD technology will be discussed in more detail, particularly with respect to future space applications.

The first part of this paper briefly presents recent developments in Ion Assisted Deposition (IAD) with respect to the type and applicability of the available sources. In addition, some innovative IAD process developments for depositions on temperature-sensitive substrates will be discussed. The final section of the paper will explore the future role of IFCAD in coatings for advance space applications.

3. DEVELOPMENTS IN ION ASSISTED DEPOSITION

The early work with IAD processing concentrated on the observed dramatic improvements in thin-film adhesion and density. With the subsequent wide introduction of end-Hall and other ion sources into the optical thin-film deposition environment, energetic ion sources have been applied in a wide variety of new and difficult applications. Most recently, the telecommunication industry boom was predicated upon the production of extremely narrow band pass filters (DWDM) that are stable under the influence of severe moisturer and temperature conditions. The DWDM filter market was fueled to over-capacity, in part, because of the successful application of energetic process technology in the production of extremely high performance coatings.

Energetic process technology is briefly summarized in this section culminating in the most recent work with pulsed IAD for the production of fluoride coatings. Historically, the ion bombardment of growing films have moved through a progression from the early Kaufman-type Gridded Ion Sources, RF Plasma Sources, Plasma-Assisted Deposition, and the Gridless Ion Sources.

3.1 Gridded Ion Sources

Without dwelling on the particulars of this type of source suffice it to say that the work done with Gridded Ion Sources by the early IAD researchers laid the foundation for the successful work of subsequent broad-beam IAD technologists.[22] These sources continue to play a major role in conventional Ion Beam Sputtering and Ion Milling applications that require high ion energy (1000 eV) and a collimated beam. In addition, these sources have been successfully used in IAD applications for relatively small are substrates (< 8 inch dia.), including DWDM filters.

3.2 RF Plasma Sources

RF and Microwave sources have had a prominent role to play in the development of advanced PECVD processes.[23] The author had the opportunity to participate in the development of an unique RF source that permitted the production of an ITO (Indium Tin Oxide) film that was fully transparent with a sheet resistance of less than 1.0 Ω/sq. over a large substrate area (1 meter dia.)

3.3 Plasma-Assisted Deposition

Two companies, Leybold and Satis Vacuum, developed a powerful presence in the application of IAD technology.[24-25] It is interesting to note that their Plasma-Assisted Deposition technologies were primarily designed for ophthalmic applications. As the telecommunication market grew, both companies adapted their technology to manufacture narrow band filters (DWDM). Without delineating the specific differences between the two technologies, it is sufficient to state that both sources operate by ionizing Argon gas in the reaction chamber of the plasma source. By the placement of an oxygen gas shower ring over the source's output opening the reactive gas is ionized by the plasma. The ensuing reactive mixture of Argon and Oxygen ions provides an IAD process for the evaporation source materials.

3.4 Gridless Ion Sources

There are a several companies producing ion sources for IAD applications. Even though the author does not have experience with all the available ion sources, it has become clear through working extensively with two end-Hall ion sources, Veeco Mark II (formerly Commonwealth Scientific) and SainTech ST2000, there are generalized source attributes that should be explored.

In the perpetual pursuit to improve the performance of thin-films produced with IAD, the sources have been modified to deliver more ion beam current and energy. The first step in this quest for higher performance IAD was the collaboration between the author and Commonwealth Scientific to create a High-Output End-Hall Ion Source.[26] This ion source was modified to accommodate a water-cooled anode that enabled a substantial increase of the anode current from five to ten Amps. In addition this source was equipped with a hollow cathode electron supply, extending the run-time (>100 hrs) over a standard single filament configuration (< 8 hrs).

It was during the development and engineering phase for the International Space Station (ISS) window coatings that a limitation for this source was reached. Due to the large substrate size (29 inches dia.) placing two High-Output Mark II sources in the chamber appeared to be a reasonable approach. However, a serious problem ensued. The two sources, in simultaneous operation, arced vigorously, creating destabilizing oscillations in the power supply feed-back control system. Evidently, there was excess gas in the chamber that provided a medium for plasma shorts between the two ion guns and e-beam evaporation source. By reconfiguring the chamber and the attending process conditions only one High-Output Mark II source was eventually used to successfully complete the required Space Station Window coatings.

Concurrent with this advanced work on the International Space Station (ISS) window coatings an investigation using the SainTech ion source technology was initiated. Motivated by the need for a higher conversion of gas to ions this source was evaluated in a chamber with a similar configuration to the original two ion source set-up used for the ISS window program. The benefits for a higher conversion of gas to ions in the IAD process go beyond the ostensible requirement of placing two ion sources in the same chamber. Pumping speed of a chamber ultimately determines the ability to handle the gas load of the ion sources. In most manufacturing environments the chambers are not equipped with pumps that can accommodate the high gas load of two end-Hall ion sources. Developing ion sources that use less operating gas is very beneficial for manufacturing high performance thin-film coatings.

The quest for low temperature deposition of metal fluorides, particularly MgF_2, has been pursued by a number of researchers.[6,27] The SainTech development of the so-called "pulsed IAD" approach has the potential to provide the thin-film practitioner with another important tool for the deposition of low index thin-films.[28] Coupled with the lower background gas environment, the "pulsed-IAD" has been successfully applied by the author to the deposition of Yttrium Fluoride as a Thorium Fluoride replacement material in Far IR applications.

Based on the author's twenty plus years experience with IAD there are certain attributes of an ion source that are desirable to maximize thin-film performance:

(1) Water-cooled anode: this is an option for many vendors; however, for higher beam currents, particularly for temperature-sensitive substrate applications, the water-cooled anode is a necessity.
(2) Extended filament life, or hollow cathode: the efficient production of electrons for the IAD source depends on the ability to switch filaments if one breaks; or the source can be equipped with a hollow cathode. None of the commercially available hollow cathode sources are water-cooled—this is a recommended feature for temperature-sensitive substrate applications as well as extending the life of the hollow cathode itself.
(3) Anode designed to maximize gas ionization: in order not to overstress the pumping systems with excess gas special attention to the design of the anode to maximize the conversion of input gas to ions is highly advantageous.
(4) Electro-magnet instead of permanent magnet: none of the commercially available ion sources offer a electro-magnet. The original Kaufman and Robinson ion source had an electro-magnet that provided the operator with another process parameter for optimizing the performance of the source.
(5) Ease of assembly: this a self-evident positive attribute of an ion source.
(6) Reliable and robust power supply: another self-evident positive attribute that has not been achieved in many of the past designs.

Even though there has been tremendous progress in the development of end-Hall ion sources over the last 20 years, there is still room for much improvement. Not only is the operational reliability of the source improving steadily, but the applications for IAD processes development is growing proportional to the expansion of the electro-optical market, particularly for space and military programs.

4. DEVELOPMENTS IN ION-ASSISTED FILTERED CATHODIC ARC DEPOSITION

4.1 Background

Sanders *et al* effectively summarized the development of arc technology, including the anecdote that Thomas Edison produced coatings using a vacuum arc over a hundred years ago.[29] Even though arc technology has significantly penetrated commercial markets, particularly in the coating of machine tools with metal nitrides to extend their lifetimes, its full potential has not been exploited.

Historically, carbon has received most of the research attention where the earliest studies found that the Filtered Cathodic Arc Deposition (FCAD) thin-film properties are highly dependent on the ion energy (usually controlled by applying a substrate bias). Additionally, the, so-called, "macro-particles" generated simultaneously with the FCAD ions, could not be removed completely by early filtering system designs.[30-31]

The following summarizes some of the technical problems that have plagued the FCAD science for the past few years:

(1) Control and reliability of the process predominantly due to the arc-spot wandering randomly over, and sometimes off, the target material.
(2) Uniformity of the FCAD over distances greater than a few inches had been difficult to achieve.
(3) Deposition of multi-layer materials has not been investigated in any significant way. Most FCAD systems have used only one arc source, which meant that only one material could be used to deposit a thin-film.
(4) Co-deposition of materials is required in some advanced thin-film applications but has been difficult to accomplish due to the close proximity of the two sources, creating interference between the adjacent magnetic fields.
(5) Anode caking and poisoning has been another major historical drawback due to the adherence of macroparticles to the anode that can arrest the arc process by increasing the 'resistance' between the anode and cathode.

4.2 Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD)

The IFCAD deposition system design couples IAD into the process, eliminating most of the historical problems that have impeded the development of FCAD into a more complete production technology:

(1) In addition to a typical time and power control strategy, monolithic photo diodes have been used to measure the light intensity of the arc-plasma from various locations in the system that correlates to film thickness.
(2) An integrated magnetic control circuit design (powered by a computerized waveform generator) makes the deflection of the plasma ion beam more linear, permitting the coating of significantly larger substrates (12 inches).
(3) The IFCAD system has provision for a thermal evaporator, or e-beam gun, as well as the four FCAD sources. Design flexibility allows for the port positions in the system to hold either FCAD or end-Hall ion-beam sources.
(4) FCAD sources can be placed on either side of the chamber to allow the deposition of materials simultaneously with minimal magnetic field interference between the two.
(5) Anode poisoning is greatly reduced by the novel use of Ion-beam Assisted Deposition (IAD). The reactive gas is directed toward the substrate, reducing the amount of insulating material build-up in the anode area.

4.3 IFCAD System

The IFCAD source, as depicted in the Figure 1, below, uses a low DC voltage (high current) supply to generate an arc on a water-cooled target. The "self-sustaining" arc vaporizes the target material generating high-energy ions, neutral atoms and particles. Ejected target ions are steered by the magnetic and electrical fields through a curved duct. A mechanical "non-line-of-sight path" filter traps the particles and neutrals leaving only a pure beam of ions to enter the chamber. Since the only heat generated by this process resides in the water-cooled cathode assembly (external to the chamber), the substrate remains close to room temperature during the thin-film deposition.[19-21]

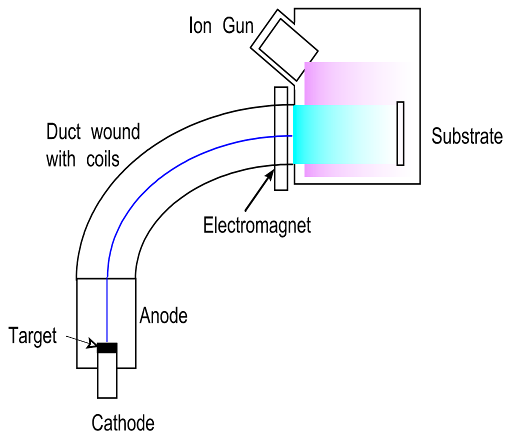

Figure I: Schematic depiction of IFCAD system

5. THE FUTURE OF IFCAD COATING TECHNOLOGY FOR SPACE APPLICATIONS

5.1 New Space Initiative

NASA's recently proposed (January 14, 2004) that the Moon will be used as a temporary rest stop for voyages to Mars and beyond. This new government initiative revitalizes earlier work done on "Lightweight Deployable Technologies" for space power and exploration. During the development of "Solar Thermal Propulsion" concepts the use of large-area deployable reflectors and concentrators were required.[32] The L'Garde company has specialized in the design and deployment of large-area space structures for solar propulsion and antennas.[33] However, one crucial challenge in the development of this technology is the inherent difficulty associated with maintaining the mechanical stability of thin-film coatings after the device deploys from an acute folded state. If the thin-film coating could be economically applied post deployment, then the performance of the film will not be compromised by environmental insults of an earth launch, or the stresses from unfolding the large flexible substrate.

Returning to the Moon provides NASA with new opportunities and challenges. The author proposes that the ability to deposit high performance thin-film coatings in the vacuum of lunar-space will be extremely valuable in executing this new mission. One immediate requirement for developing space-based thin-film deposition technology is the apparent need for the inhabitants of a lunar station to be protected by thermal control structural and window coatings. Another exciting application for spaced-based thin-film deposition technology is the coating of large-area space telescope mirrors that could be installed on the dark side of the Moon. If the Hubble Telescope is indeed scrapped, then an astronomical observatory on the surface of the Moon would be a tremendous scientific asset. Once assembled on the lunar landscape, the large-area reflectors can be coated with pure silver, for example, that will not be contaminated, corroded, nor degraded as observed in coatings produced by earth-based technology.[34]

Thin-film reflectors are of particular interest to the solar space propulsion effort, sponsored primarily by the Air Force and NASA. This technology relies on using large area coated polymer structures to reflect and focus concentrated solar energy to a space thruster. The solar thermal thruster is a high performance space vehicle suitable for transferring payloads from low earth orbit (LEO) to geosychronous orbits (GEO). One vehicle design plans to use large inflatable parabolic concentrators to focus sunlight onto a high temperature absorber.[35] This concept would be insured a greater success if the coating was deposited on the reflector after it is deployed.

5.2 Extreme UV Mirrors

EUV mirrors are of particular interest to NASA for space-based astronomical instrumentation. Considerable work has been done in this area at Goddard Space Flight Center (GSFC) where multi-layer coatings with high reflectance in the spectral range of 50 to 121.6nm have been produced and measured.[36] Amorphous Diamond-Like-Carbon (A-DLC) produced by Filtered Cathodic Arc technology has shown great promise in this application, measuring a reflection of about 40% at 74nm wavelength. The ability to use the IFCAD system for multi-layer production of EUV and other coatings hinges on the advanced film properties achieved. Carbon, for example, has been one of the most interesting materials deposited using FCAD. The thin-film properties of A-DLC are summarized in the following table:

Property	**Value**
Hardness	> 70 GPa
Young's Modulus	> 700 GPa
Coefficient of Friction	< 0.1
Critical Load	> 5mN
Percentage of sp^3 Content	> 85%
Plasmon Peak Position	> 30 eV
Density	> 3.25 g/cm^3
Stress	~ 6-10 GPa
Refractive Index	~ 2.6

Figure 2: Table of diamond-like-carbon produced with IFCAD

Using these superior properties of the A-DLC in multi-layer combination with another IFCAD material, aluminum, even higher reflections in the EUV are anticipated. The main advantage for using this thin-film combination is that it provides a much more stable coating than can be achieve using other established EUV materials and processes.

5.3 Protective Space Coatings

Protective coatings are of prime interest for solar power concentrator arrays and ultra-light inflatable Fresnel lens solar concentrators.[37] The author had the opportunity to work on Boeing's program for developing concentrator arrays using silicone Fresnel lenses. Deep Space I, launched on October 24, 1998, used solar concentrator arrays that were based on the architecture developed earlier at The Boeing High Technology Center. Since silicone degrades in the presence of UV in the space environment the Fresnel concentrator needs protection from this radiation in order to maintain the high efficiency of the lens for the lifetime of the mission. The Deep Space I mission uses silicone Fresnel lenses covered with a thin cerium doped glass to absorb the UV. However, it was demonstrated that silicone Fresnel lenses could be coated with a dielectric thin-film reflector to protect the lens from UV. The major advantage for using a thin-film coating substitute for the glass is significant payload weight savings.[38] The thin-film UV blocking design developed by Boeing for the Photovoltaic Array Space Power (PASP) module was deposited directly onto the silicone Fresnel lenses. This concept was successfully tested in space.[39]

Work done by the author this year, using a specially configured IAD process for this application, is yet to be published. Fresnel silicone lenses (DC93-500) have been successfully coated and tested for UV degradation. As of this publication of this paper the testing is not complete, but the preliminary results to date are excellent. After 1000 ESH (Equivalent Sun Hours) of VUV radiation exposure, the Fresnel lens exhibits negligible change in optical performance. This coated Fresnel lens has also been subjected to 200 thermal cycles (-170^o C to $+70^o$ C) without any apparent lost of optical or mechanical performance. In the future, the use of concentrators for space power applications could offer a significant technical advantage over conventional flat panel arrays. The coating developed for UV protection of Fresnel silicone lenses could also be applied after deployment in space on other UV radiation sensitive surfaces.

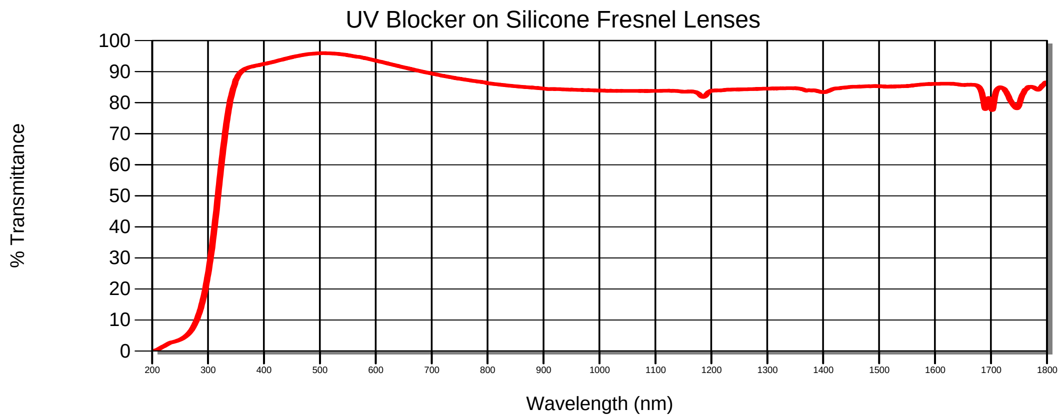

Figure 3: Measured spectrum of silicone (DC93-500) Fresnel lens, providing UV blocking and high transmission

5.4 Thermal Control

Thermal blanket coatings have been used from the beginning of the space program to protect the space vehicle from UV and IR radiation. The author had the opportunity to participate on the Boeing team which investigated the results from the Long Duration Exposure Facility (LDEF). LDEF was a satellite that spent 69 months in space to test a huge assortment of materials and coatings for space applications.[40]

Kapton, and silicone were two of many materials exposed to Atomic Oxygen (AO) and UV radiation for the duration of the flight. Coated and uncoated Kapton and silicone samples were evaluated and compared for relative degradation after exposure to the high fluence of UV radiation and AO on the prolonged LDEF space flight. The study was comprehensive, involving a number of research groups examining hundreds of samples, resulting in a voluminous set of documents. For our purpose here, a very brief description of some results is offered to provide a context for future work. The IFCAD technology can be utilized in the development of advanced coatings for spacecraft thermal blankets and other spacecraft applications.

The Kapton samples that were coated with ITO did not fare as well as the ones coated with SiO_2. Silver coated Kapton samples were also significantly degraded during the LDEF space flight. Therefore, it would be best to overcoat silver with either SiO_2 or Al_2O_3—this will be one of the approaches that the IFCAD technology could effectively address. Kapton is the preferred material for solar blanket applications. Silicone, however, provided a very intriguing result for the space community to evaluate. It appears that uncoated silicone reacts with the AO and develops a protective layer of SiO_2 on the surface that slows further degradation. Therefore, when developing a multi-layer UV reflecting coating for silicone material (See section 5.3), it would be best to terminate the design in a SiO_2 layer to insure that the lens is resistant to AO attack.

Depositing protected metal thin-films is one of the major strengths of the IFACD technology. Aluminum, silver, and gold can be deposited and protected in the same vacuum process with Al_2O_3 or SiO_2. This application would also be ideal for space-based deposition of solar blanket materials. Even the exterior of a spacecraft, or the sections of the space station, could be coated directly with a protected metal film for UV and IR protection.

6. CONCLUSION

With the emergence of energetic technologies the deposition of thin-film coatings has become one of the major driving forces in electro-optical industry. New material combinations and properties are availing themselves for a host of challenging applications in the commercial, military, and space arenas. System engineers are beginning to incorporate the results from the work done with the new energetic deposition processes in the future design of instruments, optics, devices, and structures for terrestrial and space environments.

Filter Cathodic Arc technology holds particular promise for depositing a variety of advanced thin-films at room temperature. By combing Ion-Assisted-Deposition (IAD) to the Filtered Cathodic Arc the deposition process may be the preferred deposition tool for many advance thin-film applications.

Space exploration and even colonization appears on the horizon and Filtered Cathodic Arc Deposition technology could play an important role in facilitating this endeavor by providing robotically deposited coatings over enormous space deployed structures for solar power and radiation protection. Spaced-based deposition of advanced materials could open a whole new era in astronomical observation, including large area telescopes on the surface of the moon.

As work progresses with these and other energetic deposition processes, it is the fervent hope of the author that scientists and engineers continuously seek imaginatively beneficial ways to apply their inventions and technology.

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