New Ion-assisted Filtered Cathodic Arc Deposition (IFCAD) technology for producing advanced thin-films on temperature-sensitive substrates

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ABSTRACT

An innovative Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) system has been developed for low temperature production of thin-film coatings. The IFCAD system employs electro-magnetic and mechanical filtering techniques to remove unwanted macroparticles and neutral atoms from the plasma stream. Therefore, only ions within a defined energy range arrive at the substrate surface, depositing thin-films with excellent mechanical and optical properties. Ion-Assisted-Deposition (IAD) is coupled with Filtered Cathodic Arc (FCA) technology to enhance and modify the arc deposited thin-films. Using an advanced computer controlled plasma beam scanning system, high quality, large area, uniform IFCAD multi-layer film structures are attained. Amorphous Diamond-Like-Carbon (A-DLC) films (up to 85% sp3 bonded carbon; and micro-hardness greater than 50 GPa) have been deposited in multi-layer thin-film combinations with other IFCAD source materials (such as: Al_2O_3) for optical and tribological applications. Rutile TiO_2 (Refractive index of 2.8 @ 500nm) has been deposited with this technology for advanced optical filter applications. The new IFCAD technology has been included in development programs, such as: plastic and glass lens coatings for optical systems; wear resistant coatings on various metal substrates; ultra smooth, durable, surface hydrophobic coatings for aircraft windows; EUV coatings for space instrumentation; transparent conductive coatings; and UV protective coatings for solar cell concentrator plastic Fresnel lens elements for space power.

Keywords: Arc vaporization, Ion-Assisted-Deposition (IAD), DLC, plastic Fresnel lenses

1. INTRODUCTION

The new manufacturing prototype Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) system consists of a cylindrical rotary deposition chamber, orientated horizontally, and two (or four) Filtered Cathodic Arc (FCA) sources each associated with an end-Hall Ion-Assisted-Deposition (IAD) ion gun. Since Silicon (Si) does not arc well due to its semi-conductor material property, the system is also equipped with a special Ion-Assisted-Deposition (IAD) SiO_2 source. By coupling Ion-Assisted-Deposition (IAD) with FCAD the development of cost effective deposition processes for applying super hard advanced thin-film materials such as: Amorphous Diamond-Like-Carbon (A-DLC); Aluminum Oxide (Al_2O_3); Aluminum Nitride (AlN); Titanium Nitrite (TiN); Titanium Oxide (TiO_2: Rutile); Indium Tin Oxide (ITO); and many others are now feasible. The advanced Ion-assisted Filtered Cathodic Arc Deposition (IFCAD) system is projected to provide a unique solution to many historical hurdles encountered in laboratory versions of FCAD technology. In addition, the new IFCAD system is designed to have the ability to deposit A-DLC, amorphous Al_2O_3, and many other materials in multi-layer thin-film structures suitable for solar optical applications. The film properties produced by IFCAD technology are superior to other processes at elevated deposition temperatures, for example: the A-DLC thin-films have a micro-hardness in excess of 50 GPa (Diamond = 100 GPa); and the amorphous Al_2O_3 films have a hardness in excess of 20 GPa (bulk sapphire is 35 GPa). The new IFCAD system is ultimately designed to be an enabling technology for many novel commercial, military, and space applications.

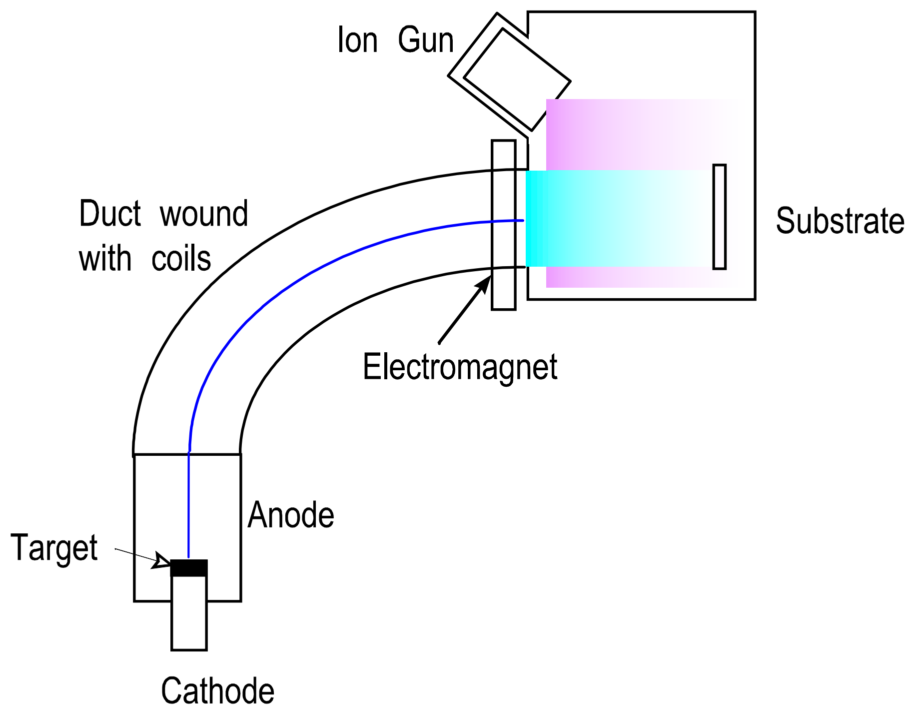

Figure 1

The above figure schematically represents the innovative IFCAD technology. A self-sustaining arc is produced in the water-cooled cathode block by a conventional arc welding power-supply (no high voltage required!). Carbon (C^+), Aluminum ions (Al^+), or other materials are ejected from the metal arc-target and magnetically steered out of the duct, while a mechanical filter captures the undesired macro-particles and neutrals. As the ejected ions emerge from the duct, an oscillating electromagnetic field scans (or sweeps) the plasma-beam to provide a uniform deposition over the substrate area. Simultaneously, a beam of gas ions, from an end-Hall ion source, impinges on the arriving arc-generated ions, resulting in dense, well adhered, stable thin-films. Due to the external location of the water-cooled arc source this deposition process is done at room temperature.

2. TECHNOLOGY BACKGROUND

More than a century ago Thomas Edison produced coatings using a vacuum arc. Nevertheless, only in the last decade has arc technology penetrated commercial markets, particularly in the coating of machine tools with metal nitrides to extend their lifetimes. Arc technology has an important potential role to play in the development of quality thin-films because FCAD effectively filters (unwanted) macroparticles leaving only highly ionized atoms that are deposited on the substrate within a controlled energy range.[1]

A small number of research institutes, and a few industrial companies, have investigated filtered cathodic arc deposition for several years. Early research work concentrated on the unique material properties of thin-films deposited using this method. Historically, carbon has received most of the research attention. Aluminum Oxide (Al_2O_3), and other materials like TiN, have more recently undergone serious laboratory investigation. The earliest studies found that the FCAD thin-film properties were highly dependent on the ion energy (usually controlled by applying a substrate bias). Additionally, it was found that these films were rendered less useful by the high density of particles adhering to the deposited coating. The, so-called, “macro-particles” were generated simultaneously with the FCAD ions, and could not be removed completely by early filtering system designs. [2-3]

Further work, therefore, concentrated on improving the removal of the macro-particles. New filtering techniques were investigated and the resultant reduction in macro-particles started to produce coatings that could be used in a number of critical applications. The new filtering designs involved changing the angles of the duct bend, increasing the substrate-to-target distance, and improving the mechanical filters.[4] The most notable recent commercial application for this technology is the coating of razor blades. Gillette has patented a specific version of the Filtered Cathodic Arc dedicated to depositing Amorphous Diamond-Like-Carbon (A-DLC) on the blades used in their new triple blade shaving system (Mach3™).[5] This gives impetus to the notion that there are many other thin-film applications suitable for FCAD technology.

2.1 Historical Drawbacks to Filtered Cathodic Arc Deposition

Even though recent research effort has concentrated on producing clean thin-films, with excellent mechanical and optical properties, much remained to be done to develop the Filter Cathodic Arc Deposition technology into a commercially useful technology. 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 was a serious obstacle to commercializing the FCAD technology. Deposition rates, although quite high (up to 5.0 nm/s $\bullet$ in^2), were often erratic. Predominantly, this was due to the arc-spot wandering randomly over, and sometimes off, the target material. It was a usual practice, during FCAD processes, to manually strike the arc, and then have the operator monitor it quite closely throughout the deposition. In addition, the arc-spot tends to erode one particular place on the target surface, exacerbating the control problem.

(2) Uniformity of the FCAD over distances greater than a few inches had been difficult to achieve. Typically, a magnetic coil placed at the exit of the bend would deflect the ion beam—a simple electromagnetic polarity change was used to sweep, or scan, the ions onto the substrate surface. However, the variation in the thickness across the substrate was not precisely controlled.

(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. However, most optical coatings require the deposition of at least two materials. In addition, it has been shown that FCAD doesn't deposit all materials well, and indeed cannot deposit materials with high electrical resistance (such as silicon). This restriction has severely limited the historical usefulness of FCA for some applications, particularly in optics.

(4) Co-deposition of materials is also required in advanced thin-film applications. Even though deposition by two separate FCAD sources, running simultaneously, has been attempted, the interaction between the two often led to uncontrolled properties in the composite film. This effect was largely 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. During the FCAD process a large amount of target material is ejected as molten droplets (or particles, in the case of materials like carbon). These particles readily adhere to the anode and can arrest the arc process by increasing the 'resistance' between the anode and cathode. Typically, the adhered target material must be mechanically scrapped off of the anode during maintenance; however, some material adheres extremely well, and cannot be removed. If the FCAD process is used to deposit materials, such as Aluminum Oxide (by converting aluminum within an oxygen background), the anode becomes coated with an insulating layer of Al_2O_3 material that will disrupt the process.

2.2 New Advanced Ion-assisted FCAD (IFCAD) System Design

Coupled with Ion-Assisted-Deposition (IAD), the advanced IFCAD (featuring a custom designed particle-free filtration) has a particularly important role to play in the advancement of electro-optical material science. Empirical evidence has confirmed that IAD substantially improves thin-film properties when compared to conventional Physical Vapor Deposition (PVD). In summary, it has been demonstrated that bombardment of a growing film with energetic ions enhances the performance of the thin-films: 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 are positively influenced by this technique, such as: residual stress modification; surface morphology structures (crystal orientation, smoothing, and grain size); enhanced optical performance (stable refractive index and low-absorption); and durability.[6-9]

Therefore, the new IFCAD deposition system is designed, by coupling IAD into the process, to eliminate most of the historical problems that have impeded the development of FCAD into a complete production technology. The following are some of the innovations that have been incorporated into the design of the new IFCAD system:

(1) It is now possible to control the FCAD process more reliably by using feedback from sensors to initiate and maintain a more consistent output from the FCA source. Monolithic photo diodes are used to measure the light intensity of the arc-plasma from various locations in the system. This feedback control system is designed to maintain a reliable deposition rate. In addition, the monitoring system will adjust the position of arc initiation over most of the target area resulting in uniform erosion of the surface.

(2) By employing a complete integrated magnetic control circuit design (powered by a computerized waveform generator) the deflection of the plasma ion beam is much more linear. The combination of one beam-scanning axis with a second substrate rotational axis results in the ability to uniformly coat large, complex, substrate surfaces. In addition, the prototype system can accommodate up to four individual FCAD sources, permitting the coating of significantly larger substrates than any single source system.

(3) The new innovative 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. The system's versatility provides for the development of advanced deposition processes, for high performance coatings, by permitting wide latitude in the selection of the best source and location for each thin-film material.

(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. The substrate holder effectively acts as a shield between the sources—preventing deleterious magnetic interaction.

(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 in the anode area. Additionally, an innovative protective shield, made from either graphite or aluminum, is used to protect the actual anode from being poisoned by target material.

3. ION-ASSISTED FILTERED CATHODIC ARC DEPOSITION (IFCAD) SYSTEM SUMMARY

Much like IAD technology in the 1970's, FCAD has been confined to the realm of academic and laboratory investigations. Building on the pioneering experience of bringing the first gridless end-Hall ion source into production for optical coating applications, a similar scenario is being pursued for the commercialization of the IFCAD technology:

The new prototype Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) coating system consists of a cylindrical rotary deposition chamber, orientated horizontally, incorporating up to four Filtered Cathodic Arc (FCA) sources and an special Ion-Assisted-Deposition (IAD) SiO_2 source. Aluminum Oxide (Al_2O_3) in combination with other materials, such as: Amorphous Diamond-Like-Carbon (A-DLC); refractory metals; metal oxides; compounds and alloys of such materials can be deposited onto various types of substrates. The deposition process involves rotating a large area cylindrical substrate holder, mounted with a variety of possible geometric substrate configurations (consisting of either metals, semiconductors, plastics, ceramics, or glass), past the FCA sources that sweep an uniform beam of deposition ions onto the rotating substrates. The FCA sources, in separate, sequential, or simultaneous depositions, synthesize a broad range of materials, <u>at ambient temperature</u>. Extremely durable advanced electro-optical thin-film materials, of nominal dielectric constants, can be reliably produced with a controlled uniform coating thickness.

Central to this innovative design, as compared with previous systems, is the ability to deposit multi-layer combinations of advanced electro-optical materials onto temperature-sensitive substrates. The quality of the Aluminum Oxide film, for example, produced by this FCA technology, is expected to exceed other Al_2O_3 films deposited at elevated temperatures in terms of hardness, Young's Modulus, density, dielectric constant, and surface smoothness. The cylindrical rotating geometry used in the system design will provide for high rate, uniform deposition of quality coatings, ideally suited for advanced tribological and electro-optical applications.

The IFCAD source, as depicted in the Figure 1, 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.

The plasma beam (ions) is horizontally scanned at the exit of the duct using an electro-magnetic field controlled by a computerized waveform generator. Analogous to brush painting, the ion beam is swept side to side to uniformly coat the substrate. A sizeable deposition width is obtained, typically greater than ten inches. When the substrates are mounted on a rotating drum, then a significant volume of components can be coated efficiently at low cost. In addition, the emerging ions can be accelerated (or slowed) by either applying a RF or DC substrate bias (depending on the substrate's electrical conductivity), permitting accurate control over the energy of the arriving ions. The arriving metal ions (Al, Ti, Ta, etc.) can be oxidized and densified using the Mark II High Output ion source (Commonwealth Scientific Corp).[6] The IFCAD process results in well-adhered, dense, uniform, and stress controlled thin-film coatings. The deposition rate for Al_2O_3, or Carbon, is greater than 5.0 nm/s $\bullet$ in^2, which compares favorably to most other conventional deposition technologies.

4. SOLAR OPTICAL APPLICATIONS

There are a number of solar optical applications that are under investigation using the new IFCAD technology, including: EUV mirrors; room temperature transparent conductors; thin-film reflectors; protective coatings for solar optics, and spacecraft thermal blanket coatings. Each of these applications is being investigated in relationship to either specific solar requirements, or in parallel with other commercial, aerospace, and military research. A brief summary of each is provided below.

4.1 Extreme Ultra Violet 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.[10] 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 this new 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. Nanoindentation, scratch test, fretting test, EELS, Raman, UV/VIS/IR spectroscopy, and surface profilometery have been used to characterize the Amorphous Diamond-Like-Carbon (A-DLC) produced by FCAD.[11] The thin-film properties of A-DLC are summarized in the following table:

Property	**Value**
Hardness	> 50 GPa
Young's Modulus	> 500 GPa
Coefficient of Friction	< 0.15
Critical Load	> 5mN
Percentage of sp^3 Content	> 80%
Plasmon Peak Position	> 30 eV
Density	> 3.25 g/cm^3
Stress	~ 6-10 GPa
Refractive Index	~ 2.6

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.

4.2 Transparent Conductive Coatings

Transparent electrically conducting films, deposited at room temperature, have many applications in the solar and display arena. The main historical constraint in the deposition of Indium Tin Oxide (ITO) is the need for substrate heating up to 300^o C. in order to achieve low sheet resistance and high visible transparency. The author has experienced some success using IAD for room temperature ITO coatings.[8] However, there is a significant differential in the melting points between Sn (232^{o} C.) and In (157^{o} C.) metals that were e-beam evaporated with IAD in the sited work. This separation in melting points (and the associated relative vapor pressures) resulted in depletion of the In metal with respect to the Sn in the e-beam crucible mixture making a repeatable process problematic. However, the IFCAD system does not melt the material; in fact, the target stoichiometry is based on the relative boiling points.[12] Research has been initiated on varying the target material mixture ratio between Sn (2270^{o} C. BP) and In (2080^{o} C. BP) in order to determine the optimum process parameters to achieve low sheet resistance and high visible transmission for room temperate depositions of ITO on temperature-sensitive substrates.

4.3 Thin-film reflectors

Thin-film reflectors are of particular interest to the solar space propulsion effort, sponsored primarily by the Air Force and NASA, that has been concentrating on using large area coated polymer structures to reflect and focus 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.[13] This concept requires that a number of technologies to mature prior to undertaking full-scale development. For the purpose of this discussion the concentrator is of prime interest because of the need for advanced thin-film coatings. Our technical challenge is to deposit an optical coating onto a large polymer concentrator that must be compactly stored (i.e., folded, which is potentially damaging to the coating). Then once the vehicle is launched into space the concentrator structure must be deployed while maintaining high concentration accuracy and optical quality. Two coating strategies are being explored, using the IFCAD technology, to meet the program requirements. The near-term approach is to develop a scalable version of the IFCAD system to coat the large polymer material in a terrestrial based vacuum chamber. The much longer-term idea is to use this technology in a space-based deposition configuration to deposit the films in the vacuum of space after the structure is deployed. If the economic and technical feasibility of this proposal is acceptable, then a space version of the source will be designed. The obvious advantage of depositing coatings in the vacuum of space is that the terrestrial environmental insults that attack coated optics are avoided. The disadvantage is the extra spacecraft weight and power required.

4. 4 Protective coatings for solar optics

Protective coatings are of prime interest for solar power concentrator arrays and ultra-light inflatable Fresnel lens solar concentrators.[14] 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, uses solar concentrator arrays that are 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 has to be protected 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 silicon Fresnel's 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.[15]

The thin-film UV blocking design that was developed by Boeing for the PASP module was deposited directly onto the silicone Fresnel lenses. This concept was successfully tested in space.[16] Late last year some additional work was done on a new plastic material, THV500. Several THV500 samples were successfully coated using IAD. However, the UV testing of the THV500 material has demonstrated that it would be preferable to coat the silicone Fresnel lenses for future missions. The actual spectral performance of the blue reflecting coating on THV500 is shown in Fig. 2.

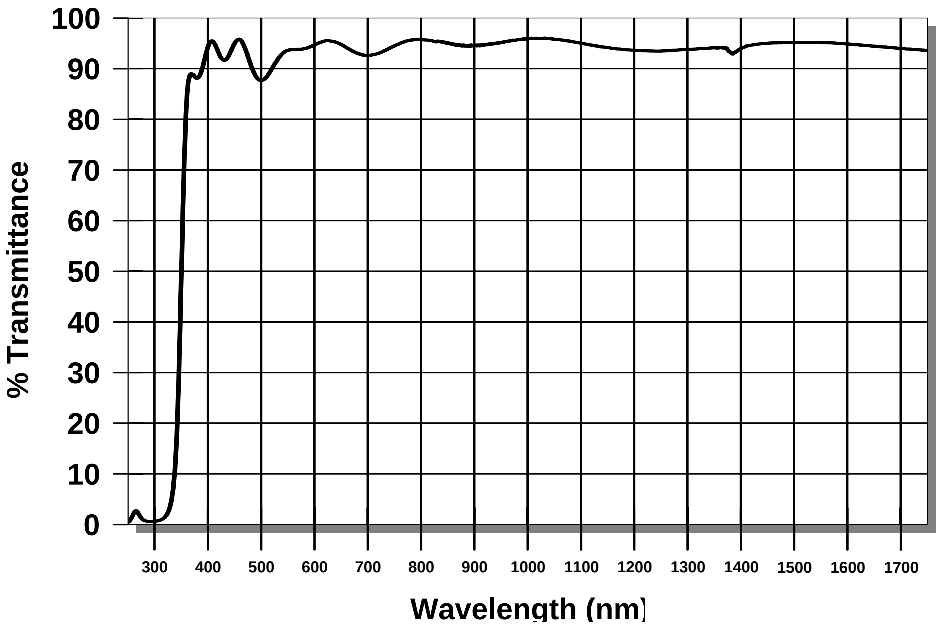

Figure 2

Entech Inc. (see acknowledgments), the developer of the concentrator molded lenses, supplied the THV500 Fresnel samples that were coated using IAD. The samples were measured and tested for UV degradation, providing results showing that silicone is a more suitable material for this application.

Future work, using the new IFCAD technology, is being planned for developing the next generation of coatings for the silicone Fresnel lenses. The fact that the IFCAD coating process can be done at low temperature substantially contributes to solving the major technical problems involving the silicone material. Intrinsic thin-film stress and the differential in thermal expansion between silicone and the thin-film material conspire to make the mechanical stability of the deposit film difficult to achieve. By varying the selection of thin-film materials and the IFCAD process parameters it is proposed that a low stress, thermally stable film, can be deposited onto the Fresnel silicone concentrator lenses.

4.4 Spacecraft thermal blanket coatings

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 who 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.[17]

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 4.4), 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.

5. CONCLUSION

The new Ion-Assisted Filter Cathodic Arc (IFCAD) holds great promise for depositing a variety of advanced thin-films at room temperature. Solar optics is of interest because the materials that are required can be effectively used in many other commercial and aerospace optical applications. In fact there is no other technique that can produce the quality of some of these materials at room temperature. This is particularly true for Amorphous Diamond-Like-Carbon (A-DLC) and Rutile TiO_2.

By combing Ion-Assisted-Deposition (IAD) to the Filtered Cathodic Arc a new deposition process is now emerging for many advance thin-film applications that will be arising as the new century dawns. Space exploration and colonization is on the horizon and this IFCAD technology should play an important role in facilitating this endeavor. 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.

Before any of these future applications can be realized a concerted research and development effort will be conducted applying the new IFCAD technology to many of the historically difficult deposition applications that have challenged thin-film practitioners.

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