

Ion Beam Assisted Deposition for Optical Coatings: R&D to Production

J.K. Hirvonen, U.S. Army Research Laboratory, Watertown, MA

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ABSTRACT

Ion Beam Assisted Deposition (IBAD) involves vacuum deposition processes which incorporate concurrent energetic ion bombardment and material deposition. The effects of the ions are to promote adhesion, densify the films, and to help chemically incorporate reactive ions into the coatings. Examples of IBAD studies that range from R&D to production status are presented with a discussion of the technical factors influencing their commercial acceptance in the optical coating community.

INTRODUCTION

The IBAD process has been studied in several R&D labs around the world since the 1970s and has been employed in several diverse application areas, including optical coatings, electronic thin films, metastable alloys, and protective coatings for corrosion and wear applications [1]. The presence of energetic ions during thin film growth has been recognized since the 1960s as an important and beneficial factor in several vacuum and plasma based coating processes. The effects of the ions are to promote adhesion, densify the films, and to help chemically incorporate reactive ions into the coatings.

Some of the earliest promoters of this process include those in the optical coating community for producing dense, non-porous, and stable optical coatings. This application typically employs evaporative or sputter deposition combined with a broad beam source of directed ions, allowing independent control of the ion and neutral vapor transport rates and the ion energy, but is limited to line-of-sight processing. This independent control by use of a separate ion source has also been an important asset in studying the role of ions in plasma processes such as ion plating.

ION BEAM ASSISTED DEPOSITION

Several aspects of film growth beneficially influenced by ion bombardment during thin film deposition include control and/or improvement of: i) adhesion, ii) nucleation or nucleation density, iii) internal stress, iv) morphology, v) density, vi) composition, and vii) the potential for low temperature deposition on temperature sensitive substrates. IBAD processing

allows the attainment of thicker alloyed regions than can be formed by direct ion implantation, and also incorporates desirable features attributable to ion beams, such as superior adhesion due to precleaning and ion mixing during the initial stages of deposition.

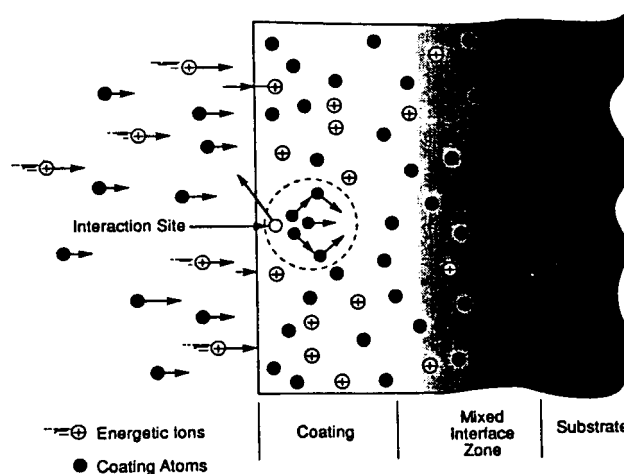


Figure 1. Schematic Diagram of Ion Beam Assisted Deposition (IBAD) Process.

Figure 1 shows a simplified schematic of the IBAD process incorporating physical vapor deposition with energetic ion bombardment. IBAD processing can be arbitrarily grouped into three different categories:

- 1) Nonreactive IBAD - where the main purpose of the ions, typically inert gas ions (Ar^+), is to influence the nucleation and growth of deposited elements or compounds.
- 2) Reactive IBAD - where the purpose of the ion beam is to both influence film growth as well as to provide atoms for the growth of a chemical compound (e.g., Si or Ti deposition with nitrogen ion bombardment to produce Si_3N_4 , or TiN).
- 3) A variation of this reactive technique is to provide atoms in the form of backfilled molecular gas (e.g., N_2 or O_2) while incorporating (i.e. activating) these atoms into the growing film by bombardment with energetic ions (inert or reactive ions). This last variation can sometimes be used to create stoichiometric compounds if (and only if) the evaporant is sufficiently reactive [2,3]. It can also be used to make up for the loss of a constituent element (e.g., oxygen) when evaporating com-

pounds (e.g., Al_2O_3 or SiO_2) which tend to decompose at high temperature giving a metal-rich coating in the absence of such an O_2 backfill.

A typical geometry of IBAD equipment is shown schematically in Figure 2. The neutral species is normally delivered via physical vapor deposition using either sputter deposition or evaporation. The ion species is typically provided by a low energy (0.2-2 keV) broad-beam gridded ion source producing beam currents up to $1\text{-}2\text{ mA/cm}^2$ (i.e., circa 10^{16} ions/sec/cm²).

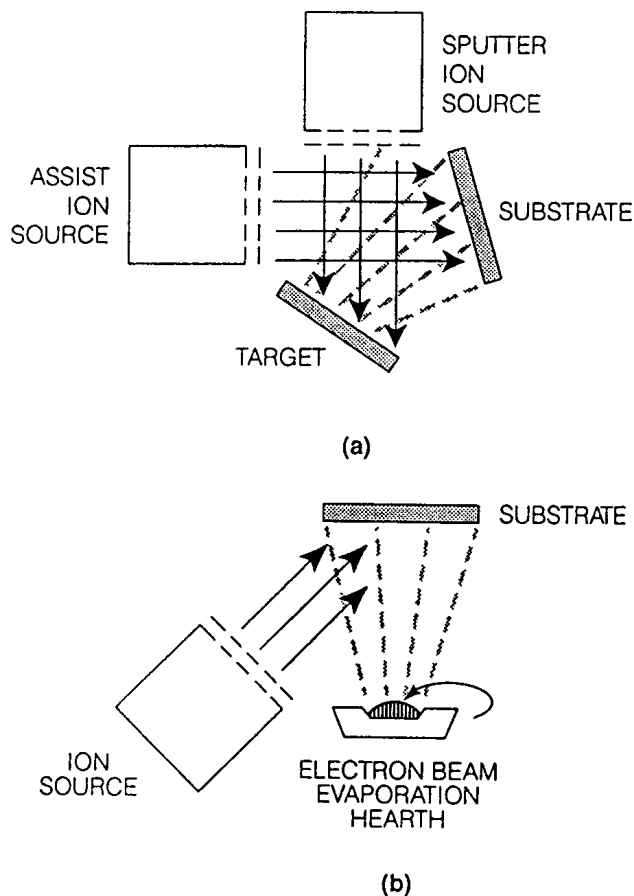


Figure 2. Schematic of geometries for energetic ion bombardment of thin films deposited by physical vapor deposition, commonly referred to as Ion Beam Assisted Deposition (IBAD) employing: a) sputter deposition, b) electron beam evaporation.

Adhesion

The adhesion of thin films on substrates depends on a variety of factors including: i) interface chemistry, ii) film stress, iii) differential thermal expansion, iv) contaminant levels at the interface, and v) surface morphology. This area has been studied extensively by Baglin [4]. *In situ* vacuum ion beam cleaning offers an excellent means of preparing substrates for coating by removing contaminant layers (e.g., adsorbed water, hydrocarbons, oxides, etc.) and sometimes by selective removal of

surface material (texturing) to leave a favorable high bonding surface for either chemical or morphology (texture) influenced adhesion.

The use of high energy ions to mix or “stitch” a metallization onto a substrate has been realized for many years. This technique requires an accelerator capable of producing ions of high enough energy to completely penetrate the film to be stitched, however, which limits practical film thicknesses to tens to hundreds of nanometers for medium mass ions of energies up to 200 keV.

Low energy ions used in conventional IBAD processing (ion energies typically less than 2 keV) also appear to be effective for enhancing adhesion. Baglin and colleagues have conducted many adhesion studies of Cu on Al_2O_3 by using high energy ions of He and Ne to stitch their films [4]. They found that the improved adhesion could not simply be attributed to a physical intermixing of Cu and Al_2O_3 . They were also able to achieve similar results at low ion energies by pre-exposing the Al_2O_3 to a low energy argon beam (500 eV, $50\text{ }\mu\text{A/cm}^2$) and then depositing the copper [5]. They found, however, that there was an optimum sputtering dose to obtain optimum adhesion. This effect was attributed to preferential sputtering of oxygen from Al_2O_3 , such that at a given composition ($\text{Al}_2\text{O}_{3-x}$), copper will strongly react to form stable ternary bonding configurations, or interface phases. XPS analysis of a sample prepared under these same conditions yielding the best adhesion showed a new line, not identifiable with known binary compounds, and this line is thought to correspond to a ternary compound. Baglin notes that Cu/ Al_2O_3 samples prepared using presputtering showed no systematic increases in adhesion after higher energy “stitching” experiments using Ne or He ions at 250 keV, implying that bombardment reconfigures interface chemical bonds.

Densification

The common occurrence of (columnar) microstructural features in low temperature coatings is well documented [6,7]. The use of energetic ion bombardment during low temperature deposition offers a means of essentially eliminating this mode of microstructural evolution. K-H Müller [8] has theoretically modeled the evolution of a two dimensional lattice under ion bombardment. Using two dimensional molecular dynamics calculations, he shows the microstructure evolving from a highly porous to a highly compact structure as a function of temperature and ion energies. Müller’s calculations demonstrated that bombardment of growing films with energetic ions removes overhanging atoms, induces surface movement of atoms, produces local heating, and collapses voids.

Stress

Stress in thin films can be related to microstructure, as well as to incorporated impurities. Typically, thin films with high void

fractions are in a state of residual tensile stress. When deposited with the aid of an ion beam, the microstructure initially densifies allowing even higher attractive interactions between adjacent atoms which can further increase the tensile stress to a maximum. As the ratio of incident ions to arriving neutral species increases, the lattice density increases further and the stress may be driven towards a state of compressive stress by the forcible injection of beam atoms or by lattice disorder caused by the energetic beam [9].

IBAD Optical Coatings

Portions of the optical coating community [10,11,12,13] have adopted ion beam assisted processing because it offers several advantages over conventional e-beam evaporation with a minimum of additions to the equipment required. Some of the advantages are listed above in Table I.

The use of ions for precleaning a substrate is used extensively in plasma based processes and is an important feature of ion beam assisted processing of optical components for enhancing adhesion by removal of adsorbed contaminants. This cleaning can be done at ambient temperatures, circumventing the requirement of employing elevated substrate temperatures normally required to gain adequate adhesion.

Table I: IBAD Processing for Optical Coatings

PROCESS ADVANTAGE	COMMENTS
-Superior adhesion	-Ions preclean substrate and promote interface bonding
-Higher density films -Higher refractive indexes -Lower physisorbed water pickup	-Ions eliminate voids/open defects, and promote compaction -Improved spectral stability -Fewer layers required
-Low temperature deposition -Ions provide reactivity -Lower reactive gas pressure required	-Energy of ions substitutes for elevated temperatures in compound formation
-Stress control	-Thicker (multilayered) structures possible than without ions
-Higher durability coatings -Environmentally compatible	-Densified microstructure more robust mechanically, greater cohesive strength
-Additional equipment requires no significant reconfiguration	-Robust ion sources available -Only minor changes in processing

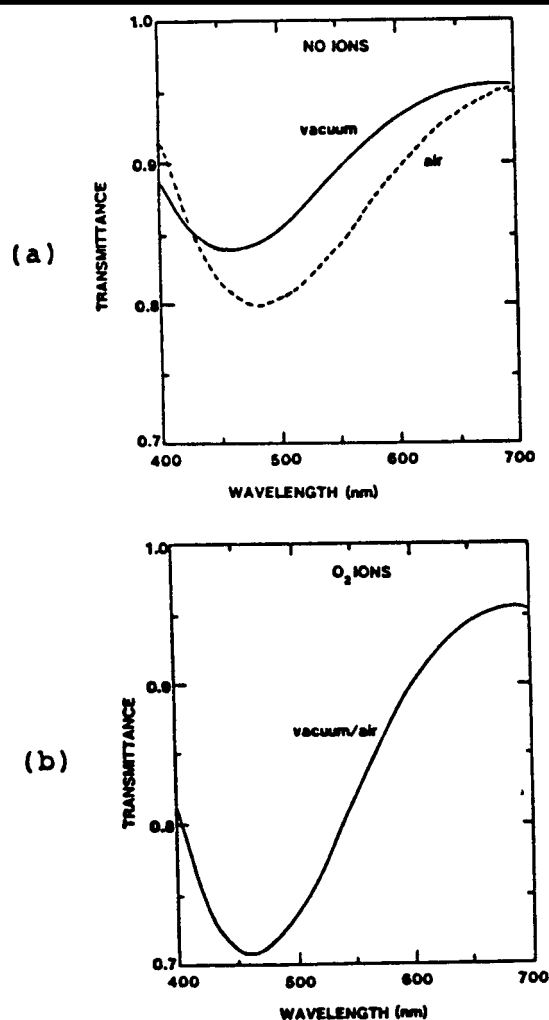


Figure 3. Optical transmittance of ZrO_2 film deposited a) without and b) with ion assistance. From Martin et al [10].

In addition to aiding adhesion, ion cleaning allows the selective removal of (partially) deposited coatings whose deposition has been interrupted or improperly done. This alone can often economically justify the inclusion of an ion source due to the extremely high cost of precision optical substrates. Figure 3 shows one of the first demonstrations of improved environmental stability achievable by the use of ion beams during deposition [14]. The bombarding ions densify the growing films as discussed above and eliminate the porous columnar microstructure normally obtained using conventional low temperature deposition. This in turn eliminates the accompanying uptake of water that occurs upon exposure to the atmosphere with the deleterious effect on the shift in transmittance as seen in Fig. 3. This problem plagued certain optical coatings prior to use of ion assisted deposition.

The enhanced reactivity afforded by energetic ion bombardment also allows for the production of stoichiometric optical coatings at lower temperatures than normally required. This allows lower (ambient) temperature processing as seen in Fig. 4 and greater choice of substrate materials, including temperature sensitive substrates.

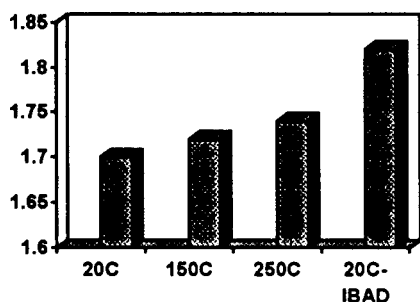


Figure 4. Refractive index (vertical axis) vs processing temperature of Y_2O_3 coatings. Data shows that ambient temperature IBAD coating has higher refractive index than 250 °C coating without ions. From Pawlewicz et al [13].

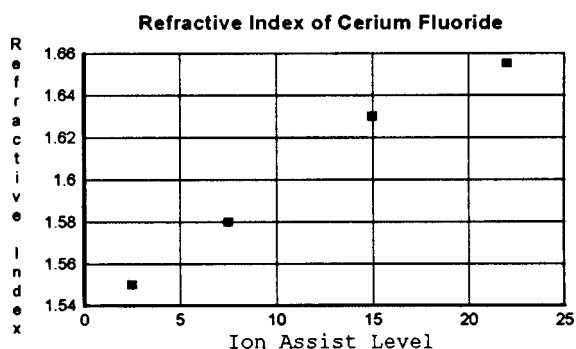


Figure 5. Increase of refractive index of cerium fluoride with increasing ion bombardment. Each unit of the horizontal axis corresponds to increments of ion assist level in units of [Amps.Volts]/[Angstroms/s]. Adapted from Pawlewicz et al [13].

The general level of ion bombardment assistance involving ion energy and ion current in relationship to deposition rate has also been used as a parameter by some in IBAD processing, as shown in Figure 5.

Table II. Improved Mechanical Properties of MgF_2 [13]

Coating Process	Abrasion Moderate (Severe)	Intrinsic Stress ($\times 10^8$ dy/cm ²)	Maximum Thickness w/o Cracking
20 °C No Ions	Fail (Fail)	50 (tensile)	0.5 microns
300 °C No Ions	Pass (Pass)	50 (tensile)	0.5 microns
20 °C With Ions	Pass (Pass)	20 (tensile)	1 micron

Another important feature of ion assisted processing is the ability to control stress levels in multi-layered coatings. Ordinarily, stress can be the limiting factor in determining the maximum usable thickness, where it can lead to delamination, as detailed in Table II. This is particularly important in multi-layered coatings (at each of the layer interfaces) and for thicker coatings such as required for IR applications. An additional related factor promoting the acceptance of ion assistance within the optical coating community is the promotion of superior abrasion resistance as a result of improved microstructure. Ion assisted coatings demonstrate higher resistance to the abrasion tests normally applied to coatings, such as rubbing with cheesecloth and pumice as two levels of severity.

These benefits accruing from the use of ion assisted deposition has made it an accepted enhancement of conventional electron beam evaporation in portions of the optical coating community requiring its benefits. Several precision coating suppliers are presently using the method on a regular basis on some of their products, but specifics concerning most commercial uses are difficult to obtain because of proprietary concerns. In any event, most of these efforts thus far have been limited to bell jar type systems.

Efforts to utilize ion assisted deposition have been significantly extended recently by Pawlewicz and coworkers [12, 13] to produce both a) larger scale optical coatings as well as b) complex, multilayer optical coatings for use in the UV, visible, and IR spectral regions. They make use of the improved optical properties obtained using ion beams as discussed above. Their 96-inch diameter chamber is 11 feet tall, with 8- and 5-foot top and bottom section diameters. It can handle optics up to 72 inches (1.8 m) in diameter and 6000 pounds in weight. It has been used for large scale optics (1 m diameter) with plus/minus 1% optical thickness uniformity over the entire substrate. Electron beam evaporation is used for the deposition of oxides, fluorides, and sulphides at rates of 0.1-0.5 nm/s. It employs a high current, low voltage ion gun with ion currents selectable from zero up to 1 A at energies of 40 to 120 eV. The ion source runs Ar, O₂, or N₂ as required with peak currents of 70 microA/cm² at the optics surface. The ion to atom ratio used is typically 0.10 to 0.20 and the energy per deposited atom is in the range of 5-30 eV/atom. The ion current density profile is precalculated using a computer spreadsheet and is optimized for the particular deposition geometry required. The ion source is used for both precleaning and assist during deposition, obviating the need for substrate heating. This greatly simplifies and reduces production costs due to i) shorter batch times, ii) less expensive fixturing, and iii) lowered substrate losses due to chipping and breakage. Ion assisted processing also allows the use of lower temperature plastic or epoxy material substrates. This facility was developed at Itek, Inc., Lexington, MA and is believed to be the largest ion beam assisted deposition facility in the U.S. It has been used since 1991 for the deposition of precision optics.

Optical coatings often serve more than one function. For example, dielectric films (SiO_2 , Al_2O_3 , or ZrO_2) used on space satellite optics are required to protect both against environmental attack (humidity) and against atomic oxygen attack. Sainty et. al. [14] investigated the relative efficacy of various dielectric layers in providing long-duration protection for Al and Ag reflection films under conditions of high humidity. Their results are shown below.

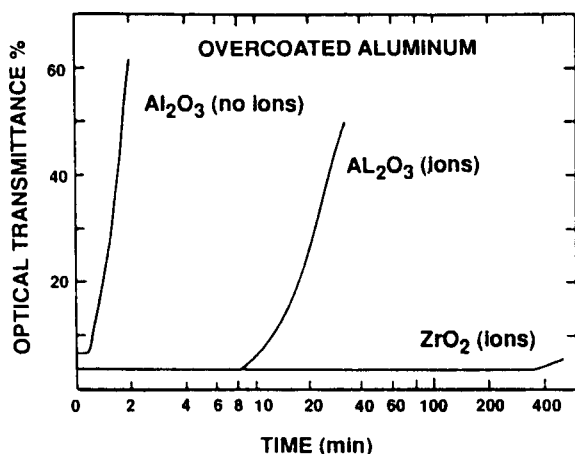


Figure 6. Corrosion resistance measurements of aluminum mirrors overcoated with dielectric aluminum oxide films. Increase of optical transmittance is due to corrosion (pinhole formation). From Sainty et al [14].

Donovan et al at NRL [15] have shown how IBAD processing can be used to make a near-infrared Rugate filter, which requires a sinusoidally varying refractive index to produce high reflection for a given wavelength. They also showed that the index of refraction for IBAD silicon-nitrogen alloys can be varied continuously from silicon ($n=3.0$) to Si_3N_4 ($n=1.9$) by suitable adjustment of the silicon to nitrogen ion flux ratios. These IBAD coatings are dense, amorphous, and avoid high tensile stresses making them environmentally stable and mechanically robust (no sharp interfaces).

Normal in-situ optical monitoring was not employed for filter fabrication, instead the IBAD process relied upon real time measurement of: i) the nitrogen ion current, ii) the silicon deposition rate, and of iii) the integrated silicon thickness. Their system was calibrated first with respect to the films' refractive index (at 6000 cm^{-1}) versus the nitrogen atom fraction in the films and secondly the nitrogen atom fraction within the films was related to the ratio of the incident nitrogen ion charge to the silicon flux rate.

During deposition of these films, Faraday cup readings and quartz crystal rate monitor readings were read into a computer which in turn controlled the extracted (nitrogen) ion current from a Kaufman type ion source to produce a sinusoidally varying composition ranging continuously in composition from Si to Si_3N_4 with other deposition parameters kept constant.

The optical transmission versus wavelength behavior of a 23 layer (sinusoidally varying composition) rugate filter deposited on Si is shown in Fig. 3. Its transmission is in good agreement with calculated values. This is another demonstration of the control of IBAD processing, making possible the fabrication of a complex optical filter previously unattainable by any other method.

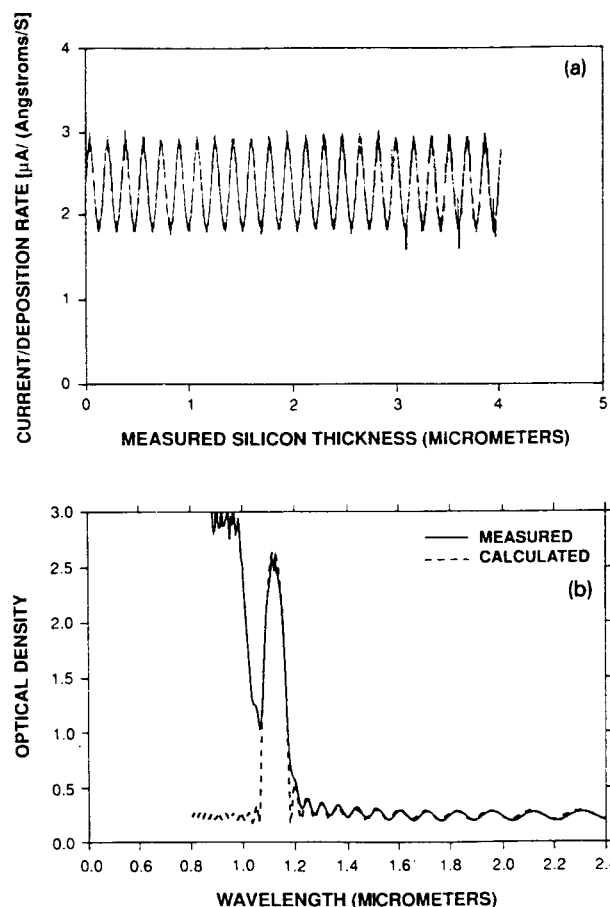


Figure 7. a) Deposition record of 23 cycle rugate filter on silicon substrate. b) Optical density vs wavelength for the filter in a) fabricated using IBAD processing. After 750 C anneal. From Donovan et al [15].

CONCLUSIONS:

Ion Beam Assisted Deposition processing has established an increasingly important role for the fabrication of precision optical coatings, due largely to its ability to control stress and porosity at low processing temperatures.

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