Properties of TiO_2 and SiO_2 Films Prepared by Ion-Assisted Deposition Using a Gridless End-Hall Ion Source

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ABSTRACT

TiO_2 and SiO_2 thin films were deposited using conventional thermal evaporation techniques with concomitant bombardment of energetic oxygen ions from a gridless End-Hall ion source. The ion-assisted deposition with oxygen ions was performed using energy levels between 40 and 120 eV and ion beam current densities of 0.3 to 0.5 mA/cm^2 impinging on the surface area. This process produces optically equivalent materials on a relatively large surface area (5024 cm^2), which makes it well suited for production environments.

INTRODUCTION

For the production of optical interference coatings a low index and a high index material are required. SiO_2 as the low index and TiO_2 as the high index material is a commonly used combination. When these materials are deposited with thermal evaporation, films exhibit poorer optical and mechanical characteristics than the bulk properties [1]. The optical and mechanical properties are dependent on substrate temperature, deposition rate and gas pressures in the evaporation chamber. These characteristics represent limitations for the optimization of the optical quality of multilayer coatings. By using ion supported process technologies, such as ion-assisted deposition, sputtering or reactive ion plating [2] the packing density of thin film coatings can be increased, resulting in an improved optical and mechanical performance of multilayer coatings. This paper will describe the production of stable SiO_2 and TiO_2 single layer films and multilayer coatings using ion-assisted deposition with an End-Hall ion source. The End-Hall ion source has been described by Kaufmann et al. [3]. It produces a broad beam (30° degree half angle) providing a uniform ion density on a rotating substrate holder or planetary type tooling (Figure 1). Moreover, the ion source has a relatively high ion current output (up to 1 A) at low energy levels resulting in improved optical properties of the deposited materials [4]. The films were deposited with thermal evaporation and simultaneous bombardment of oxygen ions generated with a gridless End-Hall ion source. This technique produced dense SiO_2 and TiO_2 single layer and multilayer films on unheated substrates.

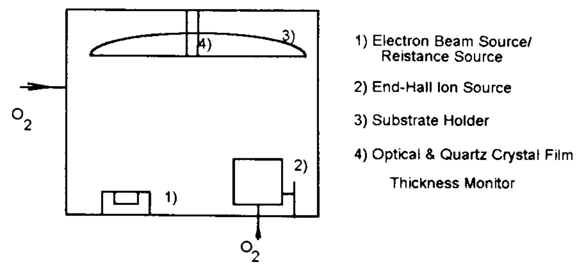

Fig. 1 Chamber configuration

PROCESS DESCRIPTION: ION-ASSISTED DEPOSITION, USING GRIDLESS END-HALL SOURCE

This process technique entails the use of a thermal evaporation source and an End-Hall source for concurrent ion bombardment of the substrate. For the formation of SiO_2 pure Si or SiO are used as a starting materials. Si is evaporated with an electron beam source, SiO with a resistive evaporation source. TiO_2 is formed by evaporating TiO_2 or a suboxide with an electron beam evaporation source. The ion beam source is operated with pure oxygen, generating energetic oxygen ions. Oxygen is also fed directly into the chamber through an additional gas inlet. This setup offers several advantages compared to standard Kaufmann type ion beam sources: 1) The

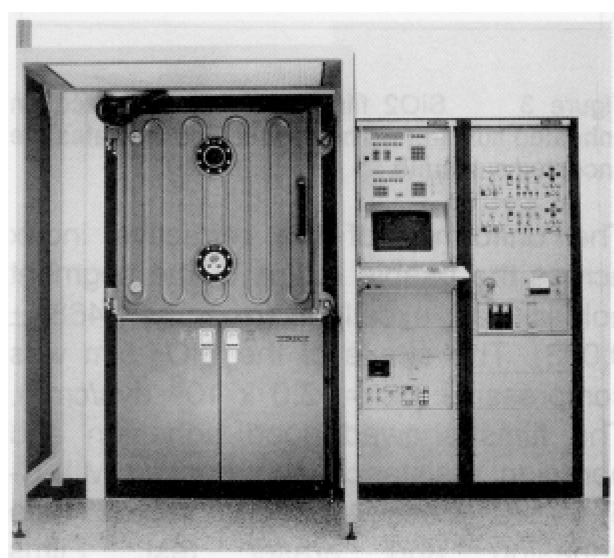

Fig. 2 Front view of BALZERS BAK 760

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ion source is emitting ions at a wide angle, rendering a uniform refractive index across the complete substrate holder. 2) The End-Hall source produces relatively high ion current densities on the substrate (0.3 to 0.5 mA/cm^2 at 400 mm source-substrate distance) [5] which leads to a significant increase in packing density of the deposited films. 3) The low levels of ion energy (40 eV to 120 eV) will cause less ion-energy induced damage than ions with higher energy produced by standard Kaufmann type sources [4].

EXPERIMENTAL SETUP

All experiments were performed in a commercial Balzers BAK 760 batch coating system, (Figure 2) configured with a 500 mm cryo refrigeration pump. The tooling consisted of a universal planetary drive with 6 planets (Ø 225 mm) or a rotating, umbrella shaped substrate holder of a diameter of 800 mm (Figure 1). The tooling type is specified with the results that are presented. The deposition of TiO_2 was performed with an electron beam source. SiO_2 was deposited using SiO as the starting material, which was evaporated with a resistance source. As an alternative, Si, evaporated with the electron beam source, was used as a starting material to form SiO_2. The ion source was a commercially available Commonwealth MARK II ion beam source. The deposition rate was monitored with a quartz crystal monitor located in the center of the substrate holder. A Balzers GSM 420 optical thickness monitor was used for optical thickness measurement and control. Figure 1 shows the arrangement of electron beam source and ion beam source in the evaporation chamber. The starting base pressure was 5.0x 10^{-6} mbar or less. Oxygen was used as the working gas through the ion source. The ion source was configured to operate at a constant gas flow. The ion current was a function of the gas flow that was introduced into the chamber. The flow was controlled by the central process control of the coating system. In addition, oxygen was fed into the chamber through a second gas inlet. The total pressure was measured with an ionization gauge. The flow through the second inlet was regulated to maintain a constant total pressure in the evaporation chamber during the evaporation. No substrate heaters were used for the deposition. All coatings were deposited with the fully automated system process control.

RESULTS

Optimizing the uniformity of the refractive index across the radius of the rotating segment holder required adjustments of the ion source position on the base plate, the source-substrate distance and the angle of the source aiming at the substrate holder. The results that are described below were achieved at an offcenter ion source position and a source-substrate distance of approximately 400 mm (Figure 1). This position produced uniform refractive indices for both coating materials that were investigated. Different locations of the ion source (while maintaining the identical ion source - substrate geometry) with respect to the electron beam source did not lead to noticeable changes of the optical characteristics. In fact, a complete separation of the ion beam and the vapor cloud of the evaporation source still produced dense, fully oxidized films.

SiO_2 Films

SiO_2 was formed by using SiO and Si as a starting material. SiO (Purity 99.9 %) was evaporated with a resistive evaporation source. Si (Purity 99.999%) was deposited using the electron beam source. The results presented below were achieved with the following parameters: SiO was used as starting material. The total pressure of oxygen was regulated to 2.0 x 10^{-4} mbar. The anode voltage was set at 150 Volts. The anode current was 3 Amperes, resulting in an ion current of 0.6 Amperes. The deposition rate was 1.0 nm/s. The resulting film had a refractive index of 1.468 at 550 nm. No absorption could be measured at 550 nm with simple photometric intensity methods (Figure 3). The films showed good homogeneity.

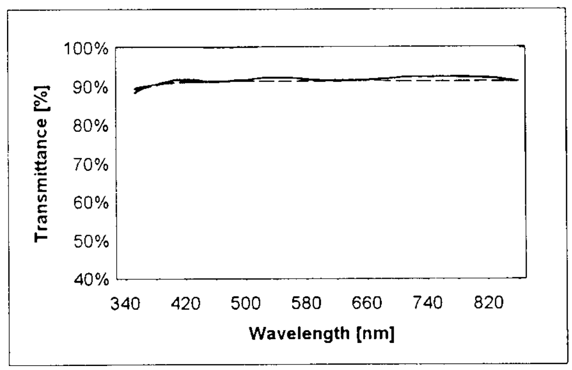

Figure 3 SiO2 film (650 nm) deposited on unheated substrate. The dashed line indicates the uncoated substrate.

The uniformity of the refractive index across the usable radius of the segment holder was excellent (n_{550} = 1.468 ± 0.003). The stress of the SiO_2 film was compressive (σ= -2.0 x 10^9 dyn/cm^2). The films showed good adhesion and abrasion resistance and passed MIL-C-675 B/C Pos. 4.5.11/12 scotch tape adhesion and eraser test. Films deposited with Si as starting material required a higher ion current (0.85 A) for a full oxidation and had a refractive index of 1.48 at 550 nm. The films were slightly inhomogeneous at a deposition rate of 1 nm/s and showed better homogeneity at lower deposition rates. Adhesion and abrasion resistance was excellent.

TiO_2 Films

TiO_2 films were formed with TiO_2 (Purity 99.9%) as a starting material. The coating material was evaporated with an electron beam source. The refractive index of the deposited film was dependent on the ion current and the deposition rate. The results described below were achieved at a deposition rate of 0.4 nm/s and at an ion current of approximately 0.92 A. The total pressure of oxygen was held constant at 3 x 10^{-4} mbar. The anode voltage was set at 150 Volts. The film had a refractive index across the usable radius of the substrate holder of n_{550} = 2.43 ± 0.02. The absorption at 550 nm was too low to be determined accurately with the simple photometric intensity method, which indicates that the absorption coefficient k_{550} was at least in the low 10^{-4} range. Figure 4 shows a TiO_2 single layer film deposited on a Corning 7059 substrate. TiO_2 films deposited with conventional reactive evaporation exhibit a highly columnar microstructure. The increase in packing density through ion bombardment leads to a crystalline microstructure according to the Thornton Zonal model [6]. An SEM analysis confirmed the crystalline modification of the microstructure. Figure 5 shows a SEM analysis of a multilayer coating of TiO_2/ SiO_2. The film stress of TiO_2 is tensile and amounts to 2 x 10^9 dyn/cm^2. TiO_2 films have excellent adhesion on glass and show good abrasion resistance. Coatings pass MIL-C-675 B/C Pos. 4.5. 11/12.

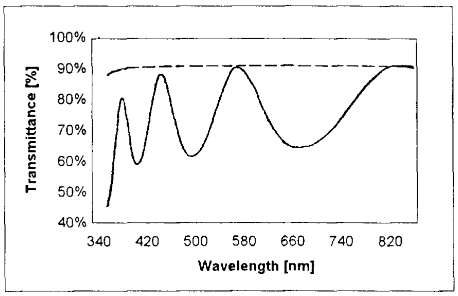

Fig. 4 TiO_2 film (350 nm), deposited on unheated substrate. Dashed line shows uncoated substrate.

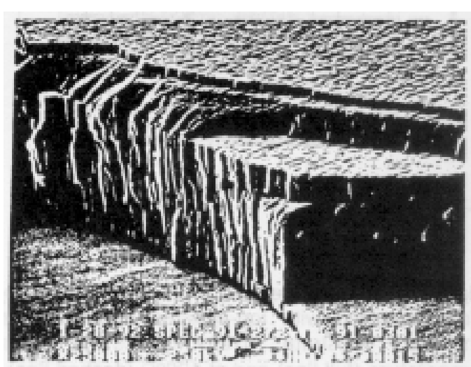

Fig. 5 SEM Analysis of TiO_2/SiO_2 film, deposited on unheated substrate.

Multilayer Coatings

Multilayer coatings were deposited using the parameter setting as described above for SiO_2 and TiO_2 single layers. No increased film absorption was observed. The high packing density through ion bombardment reduces the absorption of moisture significantly. While conventionally evaporated coatings show a shift of 6-12 nm for a temperature change from 20°C to 120°C [7], a shift of only approximately 2 nm was measured for a comparable coating produced with the described technique (Figure 6). Film stress of a 23 layer TiO_2/SiO_2 coating was compressive and, as expected, lower than the stress of the single layer films (σ = -2 x 10^8 dyn/cm^2). The coatings showed good adhesion and abrasion resistance and passed MIL-C-675 B/C Pos. 4.5. 11/12 scotch tape adhesion and eraser test.

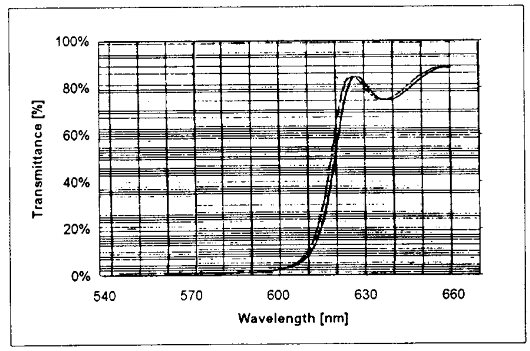

Fig. 6 Temperature shift of 23 layer TiO_2/SiO_2 film, deposited with ion-assisted deposition on unheated substrate: Transmittance at room temperature (full line) and at 120°C (dashed line).

CONCLUSION

The described setup is well suited for the production of high quality optical interference coatings. The films have increased packing densities compared to films deposited with reactive evaporation. The coatings show low absorption levels, which indicates that the films are fully oxidized and little ion energy induced film damage occurs. SiO_2 can be formed by using SiO or Si starting materials with this technique. SiO produced films with better homogeneity at high deposition rates. The uniformity of the refractive index for SiO_2 and TiO_2 can be optimized to less than ±1% across the total surface area for rotating segment dome tooling and universal planetary tooling.

While the stress level of the individual films is relatively high, the stress of SiO_2/TiO_2 multilayer films is lower, because the tensile stress of TiO_2 compensates for the compressive stress of the SiO_2 layer. Low stress is advantageous especially for thin films coatings consisting of many quarter wave stacks of SiO_2/TiO_2. Only scotch tape adhesion and eraser abrasion tests were performed. But, the high packing density is an indication that these films will be compatible with other environmental durability specifications.

IAD with the End-Hall ion source will result in shorter process cycles than conventional evaporation, because no heat and soak cycle is necessary prior to the thin film deposition. The cool-down cycle after the deposition will also be shorter, which further increases the throughput. The deposition rates are comparable to conventional evaporation.

The described deposition method is promising for temperature-sensitive applications. The principle source of heat is radiation produced by the electron beam source and the ion source. The radiation of the e-beam source can be minimized by reducing the surface area of the crucible. A cooling circuit to the ion source will further reduce the substrate temperature.

The End-Hall ion source for use in IAD processing of optical coatings has great potential. Further optimization of the described properties and for other metal oxide materials should be pursued. In addition, scaling to larger coating plants is feasible. A scale-up will require, however, an ion source with higher ion current output to achieve similar thin film characteristics using the described operating parameters.

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