Ion assist can boost the environmental stability of refractory oxide thin films deposited by physical vapor deposition.

Modified box coaters can improve oxide coatings

Hrvoje Zorc, Robert Johnson Jr., Kelly Snowdon, and Steven Morin

In optoelectronic devices such as dense wavelength-division multiplexers and optical genetic sequencers, there is a drive toward higher spectral channel density—more isolated spectral channels per unit wavelength. In instruments and systems that rely on optical thin-film coatings for spectral discrimination, the burden of supporting this increased bandwidth falls on the optical coatings and ultimately the equipment and processes that produce them.

To achieve the desired high spectral-channel density, coating equipment and processes must be developed to deposit high-phase-thickness multilayer coatings (greater than 100 layers) that realize the low temperature coefficients and the concomitant stability of the bulk refractory oxides. The realization of bulk properties in the nonequilibrium microstructures that characterize thin films is not trivial. We have retrofitted a conventional coating system with an ion gun and have tested its effectiveness in producing stable tantalum pentoxide (Ta_2O_5) films, as well as stable titanium dioxide (TiO_2) and silicon dioxide (SiO_2) multilayer coatings.

The problem

Dielectric films are often susceptible to moisture-induced spectral shifts due to their columnar microstructure. These solid columns of material extend in the direction of film growth. These are interspersed with voids running through the film thickness. This microstructure is especially pronounced in refractory oxide films deposited via conventional electron-beam-gun-based physical vapor deposition (EBPVD). In the process, an electron-beam gun generates a vapor stream that condenses onto substrates heated to an elevated temperature, typically 300°C.

These under-dense films display large (1%–5%) moisture-induced spectral shifts. A newly deposited under-dense film will shift to longer wavelengths due to the condensation of water vapor into the film voids. The magnitude of the shift depends dynamically on the environmental relative humidity

HRVOJE ZORC is with the Rudjer Boskovic Institute in Croatia. ROBERT JOHNSON JR. is president and technical director, KELLY SNOWDON is an associate thin-film scientist, and STEVEN MORIN is director of research, development, and engineering at Omega Optical, Brattleboro, VT 05302; smorin@omegafilters.com.

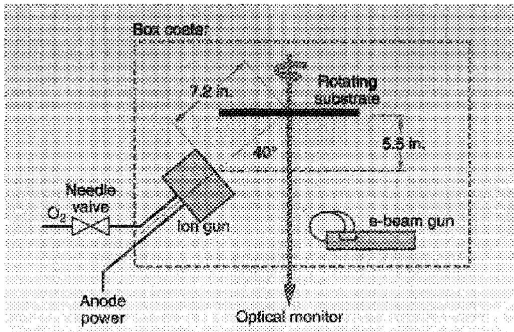

FIGURE 1. In addition to the box coater retrofitted with an ion gun, the coating equipment included a direct optical monitoring system that illuminates the geometrical center of the rotating substrate to control the optical thickness of the films. The monitoring light was nearly monochromatic and centered at 550 nm.

and thus causes problems in spectral stability of the coating. Near-bulk- or bulk-density films are devoid of significant void volumes and come closest to displaying the optical constants and stability of the bulk material. A temperature change can also cause under-dense films to experience large spectral shifts due to the impact of film temperature on the quantity of adsorbed water vapor.

The equipment retrofit

To study ways to develop refractory oxide coatings with minimal spectral shift, we recently retrofitted a conventional 1-m^3 box coater with a Denton Vacuum (Moorestown, NJ) CC-102R ion gun (see Fig. 1). All films were deposited from an electron-beam gun source onto a rotating substrate heated and soaked to 200°C. The base pressure of the heated and soaked coater was 8×10^{-5} torr. Oxygen gas was used to form the ion beam. The ion gun ion current (current density at substrate) and ion voltage (ion energy) were optimized to produce minimal spectral shift and optimal optical constants.

To study the films deposited, we constructed an environmental test chamber that fit into the sample compartment of our spectrophotometer. With this apparatus, we could monitor the spectrum of the film during changes in environmental conditions. In a typical test, the spectrum of a film was measured first at atmospheric conditions of approximately 25°C and 30% relative humidity (wet conditions). A second mea-

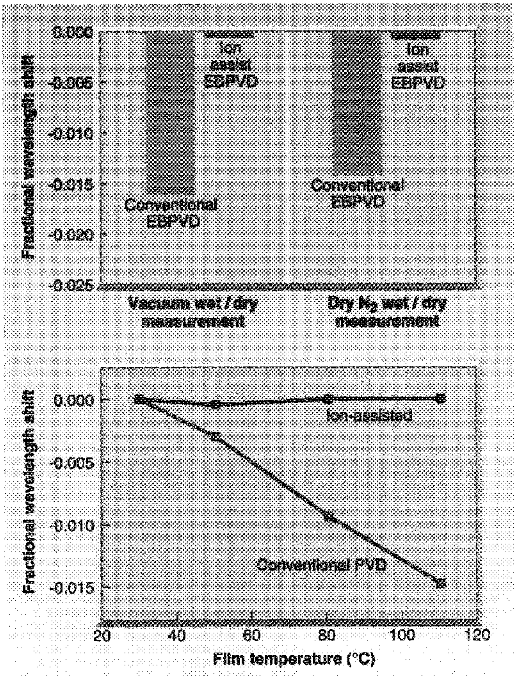

FIGURE 2. Tests of spectral shifts of a conventional EBPVD thin film and an ion-assisted EBPVD thin film under both wet and dry conditions show that a dry N_2 purge will dry a film out significantly (top). The results indicate that the film deposited with ion assist had greater stabilization against relative humidity fluctuations. Both films are Ta_2O_5 and have an optical thickness of one wave. In the dry measurement of the temperature coefficient of shift, the film deposited with ion assist displays significantly less spectral shift (bottom).

surement of the spectrum was performed with the film heated to 100°C while under a rough vacuum of 200 mtorr (relative humidity < 0.6%; dry conditions).

The results

These tests provide a good indication of the maximum shift to expect from a film because the majority of shift occurs between 0% and 20% relative humidity. These tests were run on both conventional EBPVD films and films produced by ion-assisted EBPVD.

The coating tests indicate that the ion gun can produce a tremendous stabilization effect on the Ta_2O_5 films produced by a box coater (see Fig. 2). We have achieved wet/dry fractional wavelength shifts as small as 0.0015 with ion-assisted TiO_2/SiO_2 multilayer filters. Future efforts focus on expanding the yield area for stable multilayer manufacturing to support volume production. ❑

This article is adapted from a technical presentation in the 41st Annual Technical Conference Proceedings, 243–247, Society of Vacuum Coaters (1998).