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Progress & Developments: OSA Optical Interference Coatings Conference

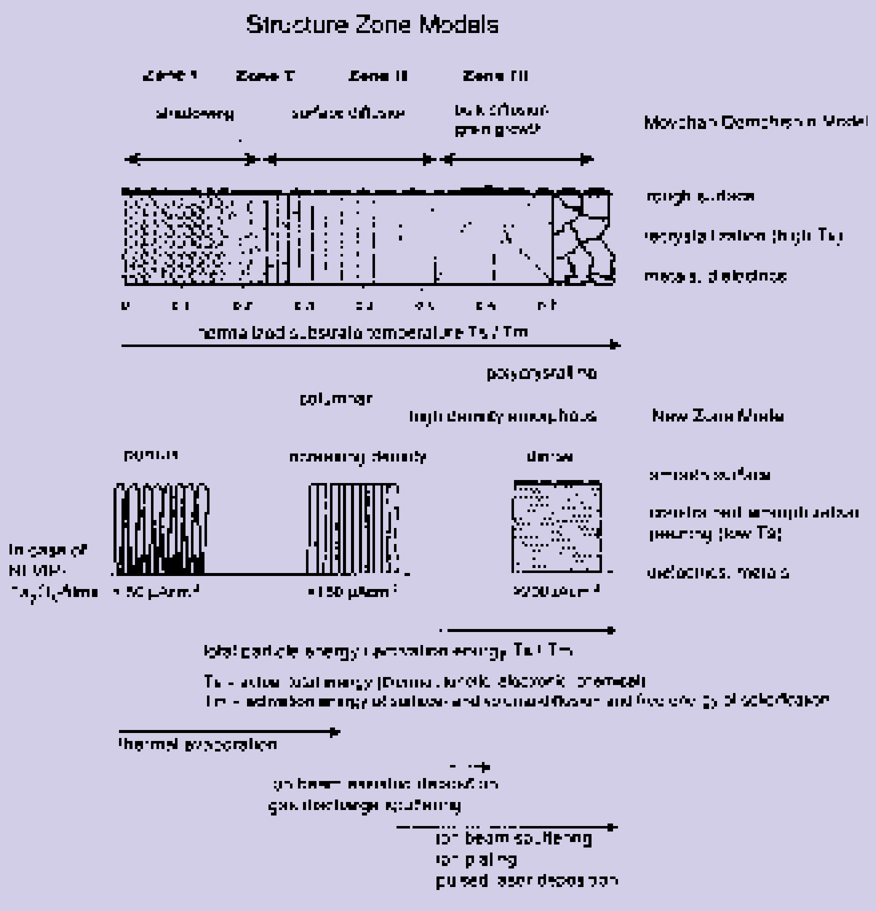
At this year's OIC Conference, held in Tucson 27 June to 2 July, an international gathering of speakers and attendees shared information on new developments and accomplished progress on a variety of topics. Not surprising, general topics were the deposition, design, and characterization of optical coatings, and the wavelengths covered ranged from the deep UV to the IR parts of the spectrum.

It has been our approach when reporting on published works from the thin film literature and conferences to extract the words of wisdom that can be useful to the reader in his/her application of thin films, whether relating to material properties or deposition techniques or applications of thin film technology. The OIC provided an excellent opportunity for this material.

We begin with an instructive work by H. K. Pulker and S. Schlichtherle of the University of Innsbruck, Austria concerning the influence of film layer structure and density on the properties: refractive index, mechanical stress, optical absorption, hardness, and permeability to gases. The goals to achieve these properties in the deposited optical film layer are: bulk-value refractive index, abrasion resistant and hard layers, low (compressive) stress, and low absorption. Stability of the properties under environmental stresses is also very important. In the case of metal oxide compounds, it is necessary to establish and maintain a constant ratio of evaporant particle to reactive oxygen particle to achieve correct composition. Another relationship to be controlled is stress vs index.

E-beam and resistance-heated evaporation are low energy processes and result in the growth of low density, low index, tensile-stressed films of low environmental stability, but relatively low optical absorption. The opposite properties, many of them desirable, can be produced by high-energy processes such as sputtering and ion-bombarding assist. Typically high compressive stresses and high absorption are present in those films. Figure 1 is a modification of the original Movchan-Demchishin structure model that used temperature as the only variable, and the new zone model that incorporates

Figure 1



particle energy effects. The new zone model illustrates how density increases with increasing particle energy as one progresses from low-energy thermal evaporation to IAD and sputtering techniques, to ion-beam sputtering and ion plating and pulsed laser deposition techniques. The structure transitions from large crystallite columns to more densely packed growth to amorphous form as energy is increased. These energetic processes do not require high substrate temperature, in fact the effects of substrate surface thermal energy are surpassed by the energies of the arriving species.

Working within the limited trade space, the authors used oxygen-rich plasmas and low arc-currents of a reactive low voltage ion plasma process to demonstrate acceptable index, stability, density. However, the absorption was not as low as was produced by subsequent air baking and the residual stress level was undesirable. More work is planned.

The deposition of coatings for VUV and DUV applications was the topic of several papers. Coatings for wavelengths below 190 nm are limited to fluoride compounds because oxides begin absorbing heavily there. Various deposition techniques have been tried, and it is generally concluded that e-beam or resistance-heated evaporation produce layers of lower absorption than the more energetic techniques such as sputtering and ion-assisted processes. This is because the higher-energy processes produce fluorine depletion, resulting in optical absorption. While the absorption is lower with thermal techniques, density, hardness and environmental stability are compromised. Addition of fluorine or SF_6 fluorination gases to a reactive plasma process can overcome that composition change.

Using Pulsed-IAD, where the ion beam is switched on and off cyclically with 1 s duration each and low power, Niederwald, Ehlers, Gunster, and Ristau of Carl Zeiss, Germany were able to approach the low absorption exhibited by thermal processes. Their MgF_2 and LaF_3 films were harder or more abrasion resistant as judged by a sand erosion test, and not as dense. The indices at 193 nm were slightly lower also. From this preliminary work, Pulsed-IAD looks like a promising technique for depositing fluoride films at lower energy.

Using thermal evaporation from a Mo boat, the stress and index of GdF_3 and MgF_2 layers were studied as a function of substrate temperature. Both materials are amorphous at temperature 150° C and obtain a polycrystalline columnar microstructure at 300° C, where their internal stresses and absorption are lowest. The high stress of the MgF_2 layers limits useable thicknesses in multi-layers. This work was reported by Ming-Chung Liu, Cheng-Chung Lee, M. Kaneko, K, Nakahira, and Y. Takano.

For photolithography at VUV and DUV the following wavelengths, are used: KrF (248 nm), ArF (193 nm), and for the next generation F^2 (157 nm). Coatings for these wavelengths need to be very smooth to reduce scatter to a minimum. Ion beam sputtering produces the smoothest coatings, therefore, a deposition process based on IBS will be capable of producing high quality coatings for these short wavelengths. The materials of interest are GdF_3 (index at 193 nm =1.7), MgF_2 (n = 1.44), and AlF_3 (n = 1.43). To prevent fluorine depletion, fluorine gas was added to Ar in the IBS work of T. Yoshida, K. Nishimoto, K. Sekine, and K. Etoh. Multi-layer AR coatings with <0.5% loss were deposited.

Other papers dealt with the development of UV coatings for free electron lasers where the challenge is damage resistance to the high energies encountered.

Magnesium fluoride is the lowest refractive index material available for use at wavelengths ranging from 157 nm to ~10 µm, therefore improvements in its deposition are constantly under investigation. We have discussed the optical absorption problem associated with high-energy processes. Fluorides in general tend to be in tensile stress, a fact that limits the thickness to which MgF_2 layers can be deposited. An investigation relating stress, microstructure and thermal-elastic properties to substrate temperature was reported by R. Thielsch, J. Hber, T. Feig and N. Kaiser of Southwall Europe and Fraunhofer Institute in Germany. Layers 50 nm thick were deposited on substrates of different materials and stress was measured over a temperature range 0 to 200°C. The total stress is composed of the film's intrinsic stress, thermally–induced stress and extrinsic stress from crystalline or volume changes or an external force. The thermal stress arises from the difference in TCE between the film and substrate and the temperature difference between the test temperature and the deposition temperature. The authors explained that at thicknesses approaching 100 nm, the film will exhibit a columnar structure whose packing density is very dependent on substrate temperature. However, at thicknesses near 50 nm, the QW thickness for 248 nm, the structure within the nucleated and transition zone is amorphous and otherwise different. Thick films deposited at temperatures above 250° C exhibit crystal structure and densities approaching 1.

Interesting results about intrinsic stress for films deposited at 50° C and 300° C were found. Films deposited above 250° C show lower intrinsic stress than those deposited at 50° C; the main stress component is thermal. The low temperature films show high intrinsic stress but low thermal stress. Stress measurements on the low substrate temperature films made during heating from ambient to 200° C indicated that their intrinsic stress levels approached the low values of the high substrate temperature films, probably because they lost water adsorbed in their porous microstructure when heated to the high temperature.

TiO_2 / SiO_2 Multilayer Deposition

Many pages in CMN have been devoted to discussion of TiO_2 layers and materials because the combination with SiO_2 provides the highest index ratio, and thereby the minimum number of layers required to achieve a desired performance in edge filters and bandpass filters. Titania layers are potentially hard and chemically resistant and find many applications. Improved starting forms, as described in the previous CMN, have facilitated reproducible titania layer deposition. However, the low-density columnar structure of these films can limit their environmental durability and stability. In critical applications where scattered light and transmission or reflection losses cannot be tolerated, the granularity of the titania layers must be reduced to an acceptable level. Silica layers grow with glassy amorphous, and therefore smooth structures. The granularity in the titania is attributed to its tendency to form polycrystalline columnar structures. Substrate temperatures above 200° C are required to complete oxidation reactions in the titania layers but these high temperatures promote large crystallite growth. It is desirable in production to reduce deposition time by using high rates but this leads to high substrate temperatures.

Albertinetti and Minden studied the morphology of the titania granules as related to substrate temperature increase due to the neutralizer filament on the ion gun [N. Albertinetti and H. T. Mindex, Appl. Opt., V35 no 28, 5620 (1996)]. Granularity and thus diffuse scatter increased as film thickness increased because the temperature continued to rise. The temperature needed to be as low as 85° C to eliminate granularity. But at these low temperatures titania films become absorbing.

IAD processes are described as "low temperature" processes, however we have encountered a contradiction to that claim, specifically when radiation from a white-hot neutralizer adds heat to the substrates. Even eliminating that source of substrate heating, there is evidence that the high energies of the ion beam as in ion beam sputtering where energies can approach 100 eV, the energy transfer can itself promote crystallization and thus scatter due to large grains. In the structure-zone model included on page 1, ion energy is seen to replace thermal energy. So how do we get around these contradictory processes in production?

An approach was described in a post-deadline OIC paper by B. Fan, M. Suzuki, and K. Tang of Japan. An RF type ion source was used instead of the Kaufman-type used by some previous workers. Their work is based on the premise that the substrate temperature can be 150° to 200° C, but the formation of scattering crystallites is prevented and the morphology is amorphous when the ion beam energy is increased to higher values than typical, namely 750 eV. The crystallization destruction rate exceeds its formation rate. Multi-layers with better transmission quality than unassisted E-beam layers were achieved.

The papers from the OIC that were reviewed here represent a small selected sampling of the variety of work reported on. Much of the work will be published in appropriate journals.

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