Effect of assist ion beam voltage on intrinsic stress and optical properties of Ta$_2$O$_5$ thin films deposited by dual ion beam sputtering

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Abstract

The optical properties and intrinsic stress of Ta$_2$O$_5$ thin films deposited by dual ion beam sputtering (DIBS) were studied as a function of the assist ion beam voltage (250–650 V). When the assist ion beam voltage was in the range of 350–450 V, the transmittance at the quarter-wave point reached its highest value (lowest absorption). The refractive index increased to 2.185 as the assist ion beam voltage increased from 250 to 350 V, but decreased as the assist ion beam voltage was further increased from 350 to 650 V.

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1. Introduction

Residual stress in films is composed of the intrinsic and thermal stresses acting in the coating plane parallel to the coating and substrate interface. The intrinsic stress results from the growth process and is primarily dependent on the deposition parameters, whereas the thermal stress arises from the mismatch in the thermal expansion coefficients between the film and the substrate [1–3]. Stress in optoelectronic devices can be affected by such device properties as the refractive index and waveguide loss [4]. In the fabrication of high quality optical coatings, obtaining the proper level of residual stress in the thin films is an important requirement. Multilayer coatings represent a major area of application of these films, and the performance of a multilayer coating does not depend solely on its optical properties, but also on its mechanical properties, for example, its stress and adhesion. Stress can distort a thick multilayer optical coating, and this can be a serious problem in optical systems, since the resulting curvature of the surfaces can give rise to a change in the refractive index and beam deflection.

The single ion beam sputtering (SIBS) deposition technique has been shown to be an excellent method to use for making low scattering coatings [5–7], because it involves an atom-by-atom (or molecule-by-molecule) transport process of high energy materials in a relatively low pressure and temperature environment. However, SIBS suffers from two main drawbacks, in that it is a slow deposition process and may produce oxide films with a stoichiometry problem. Therefore, in this study, we attempted to improve the film stoichiometry by mixing oxygen into the working gas (Ar) in the assist ion source [8], which is referred to as a dual ion beam sputtering (DIBS). The major benefits of the DIBS process are the increased packing density of the deposited films which makes them more bulk-like, the improved adhesion resulting from the mixing of the materials at the interfaces between each layer, and the reduction of the high tensile stress in the layers [9]. In this work, the variation in the quality of the Ta$_2$O$_5$ film was studied as a function of the assist ion beam voltage. The relationships between the assist ion beam voltage and the optical properties (transmittance and refractive index) are discussed. Also, the intrinsic stress and the change in the surface morphology of the Ta$_2$O$_5$ thin film were investigated as a function of the assist ion beam voltage used in the DIBS process.

2. Experiments

The optical quality of thin films strongly depends on the conditions used for their deposition. For the sake of comparison, we systematically studied the influence of the main deposition
parameters on the quality of the thin films deposited using DIBS as a function of the assist ion beam voltage. We obtained Ta$_2$O$_5$ thin films on an Si(111)-plane wafer using a Veeco Ion Tech SPECTOR system. Kaufman-type ion sources were used for the DIBS process. The beam voltage and current were 1250 V and 600 mA, respectively, for the first sputtering ion source. Argon gas was used as the working gas and oxygen gas was fed into the system near the target. The base pressure and working pressure were approximately 4.00 × 10$^{-5}$ and 2.67 × 10$^{-2}$ Torr, respectively. A mixture of oxygen and argon was fed into the assist ion source. Fig. 1 shows a schematic diagram of the dual ion beam sputtering (Veeco-Ion Tech SPECTOR) system.

The most frequently used stress ($\sigma$)–curvature (1/$R$) relationship used to convert the curvature to stress is the Stoney equation [10], which can be written as:

$$\sigma = \frac{1}{6E(1-v)} \frac{t_s^2}{t_f R}$$  \hspace{1cm} (2-1)$$

where $1/E(1-v)$ and $t_s$ are the biaxial modulus and thickness of the substrate, respectively, and $t_f$ is the thickness of the film. In the current study, we mainly concentrated on investigating the influence of the assist ion beam voltage on the properties of the Ta$_2$O$_5$ films based on the process conditions given in Table 1.

The analytical tools used to characterize the Ta$_2$O$_5$ films were UV spectroscopy, X-ray photoelectron spectroscopy (XPS, HP 5950B ESCA spectrometer) and atomic force microscopy (AFM). Using these techniques, the transmittance spectrum, binding structure and surface morphology were observed for the deposited thin films, respectively. The refractive index ($n$) was calculated from the ellipsometry measurements, which were based on an analysis conducted at 1550 nm.

### 3. Results and discussion

Fig. 2 shows the transmittance spectra of the Ta$_2$O$_5$ thin films deposited using different assist ion beam voltages. The transmittance spectra of the quarter-wave point shifted to a shorter wavelength as the assist ion beam voltage was increased from 250 to 550 V. When the assist ion beam voltage was in the range of 350–450 V, the transmittance at the half-wave point reached its highest value. This means that under these conditions the deposited film has the lowest absorption. Fig. 3 shows a plot of the refractive index as a function of the assist ion voltage. Initially, the refractive index increased to 2.185 as the assist ion beam voltage was increased from 250 to 350 V, but then decreased as the assist ion beam voltage was further increased from 350 to 650 V. This result can be explained by the collision between the resputtered, condensed atoms

![Fig. 1. Schematic diagram of the dual ion beam sputtering system equipment.](image1)

![Fig. 2. The transmittance spectra of the Ta$_2$O$_5$ thin films at different assist ion beam voltages.](image2)

![Fig. 3. The refractive index of the Ta$_2$O$_5$ thin films as a function of the assist ion voltage.](image3)

### Table 1

<table>
<thead>
<tr>
<th>First ion beam</th>
<th>Assist ion beam</th>
<th>Substrate temperature [°C]</th>
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<tr>
<td>1250</td>
<td>600</td>
<td>18</td>
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and the incoming atoms at higher beam voltages, leading to the decrease in the density and the refractive index of the deposited film.

A single layer of Ta₂O₅ dielectric film was deposited on a 300 μm thick Si-wafer substrate. The thickness of this single layer film was about 1 μm. The stress was measured for films deposited at various assist ion beam voltages ranging between 250 and 650 V. Fig. 4 shows the compressive stress of the deposited Ta₂O₅ thin film as a function of the assist ion beam voltage. In all cases, the film was found to be in a state of compressive stress, with a stress value ranging from 0.9481 to 0.1834 GPa. The highest stress was found for the film deposited at 250 V. As the assist ion beam voltage was increased, the stress decreased, attaining a minimum value of 0.1834 at 650 V. There is no fully accepted model for the evolution of stress in thin films. However, Davis [11] and C. C. Lee [12] proposed a simple model to explain the formation of compressive stress in thin films deposited by ion bombardment. In this model, the magnitude of the compressive stress is strongly dependent on the ion energy, with the form of the energy dependence being determined by the normalized flux \( j/R \), where \( R \) is the net depositing flux and \( j \) is the bombardment flux. This model can be expressed as follows:

\[
\sigma_{\text{max}} \propto \left( \frac{j}{R} \right)^{7/10}, \quad \sigma \propto \frac{Y}{1 - v R/j + kE^{5/3}} \quad (3 - 1)
\]

where \( E \) is the ion energy, \( k \) is a material-dependent parameter, and \( Y \) and \( v \) are the Young’s modulus and Poisson ratio, respectively. In this model, the net stress results from the competition between the stress caused by the knock-on implantation of atoms in the film below the surface, on the one hand, and the relaxation induced by thermal spike excited processes, on the other hand. In the present study, it can be seen that the ion energy was sufficiently large to reduce the stress, because increasing the assist ion beam voltage reduced the film density and refractive index.

Fig. 4. The compressive stress of the deposited Ta₂O₅ thin films as a function of the assist ion beam voltage.

Fig. 5. XPS analysis of the Ta₂O₅ thin films for a variety of assist ion beam voltages.

Fig. 6. AFM analysis of the Ta₂O₅ thin films (a) rms roughness as a function of the assist ion beam voltage, (b) AFM image at 550 V and (c) AFM image at 600 V.
Fig. 5 shows the XPS analysis of the Ta$_2$O$_5$ thin films for a variety of assist ion beam voltages. Spectral peaks corresponding to the Ta 4f$_{7/2}$ and 4f$_{5/2}$ energy levels were observed for the deposited thin films. Increasing the assist ion beam voltage did not affect the binding energy of the Ta 4f$_{7/2}$ level, because it changed the range of binding energy at which the Ta$_2$O$_5$ films were formed from 26.849 to 26.709 eV. This range of binding energies is higher than that of Ta metal (Ta 4f$_{7/2}$: 21.6 eV, 21.8 eV) [13], and is similar to that of Ta$_2$O$_5$ [14]. This means that the Ta atoms in the thin films are positively charged relative to that of Ta metal, due to the formation of direct bonds with oxygen.

The surface roughness is an important causal factor of optical scattering. Fig. 6(a) shows the AFM analysis of the Ta$_2$O$_5$ thin films for a variety of assist ion beam voltages. The rms roughness decreased to 0.1541 nm as the assist ion beam voltage was increased from 250 to 550 V, before increasing to 0.7891 nm as the assist ion beam voltage was further increased to 650 V. The surface morphology improved, showing an increase in density, as the assist ion beam voltage increased from 250 to 550 V. The surface morphologies of the Ta$_2$O$_5$ thin films deposited at assist ion beam voltages of 550 V and 600 V are shown in Figs. 6(b) and (c), respectively. The surface roughness was high, which might be due to the collision of the incoming atoms with the resputtered atoms, resulting in the deposited atoms being randomly condensed or causing significant damage to the surface when the assist ion beam voltage was more than 550 V.

4. Conclusion

Tantalum penta-oxide (Ta$_2$O$_5$) thin films were deposited by dual ion beam sputtering (DIBS) and their properties were evaluated as a function of the assist ion beam voltage. In each case, the film was found to be in a state of compressive stress at a level ranging from 0.9481 to 0.1834 GPa. As the assist ion beam voltage was increased, the stress decreased to attain a minimum value of 0.1834 at 650 V, but the binding energy was not affected. A decrease in the refractive index with increasing assist ion beam voltage was observed at higher voltages. The rms roughness decreased as the assist ion beam voltage was increased from 250 to 550 V, but increased rapidly when the assist ion beam voltage exceeded 550 V, possibly due to the collision of the incoming atoms with the resputtered atoms, resulting in the depositing atoms being randomly condensed or causing significant damage to the surface. Based on the results of this experiment, it can be concluded that increasing the assist ion beam voltage within a certain range results in a decrease in the intrinsic stress and surface roughness of Ta$_2$O$_5$ thin films.

References