

MECHANICAL AND OPTICAL PROPERTIES OF THIN FILMS OF TANTALUM OXIDE DEPOSITED BY ION-ASSISTED DEPOSITION

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ABSTRACT

Thin films of tantalum oxide 1 μm thick were deposited by oxygen ion-assisted electron-beam evaporation onto silicon and glass substrates. The packing density and optical properties of the films were controlled by the degree of ion assistance during growth. The films were characterized for hardness by ultra-micro indentation measurement and density by RBS. A strong correlation of hardness, and optical properties was found with the ion-to-vapor arrival ratio at the substrate during deposition. Evaporated films without ion assistance were found to be relatively soft with hardness values of 5.3 GPa and 6.5 GPa on glass and silicon respectively when measured with a Berkovich indenter. When the ion bombardment was increased the film hardness increased to a maximum hardness of 10 GPa for 1000 eV O_2 ion bombardment. The measured microhardness of the films was found to be influenced by the nature of the substrate for all depositions with higher microhardness values being recorded for films deposited onto silicon. The films were found to be amorphous and homogeneous. Additional ultra-microhardness measurements with a spherical tipped indenter enabled the depth dependence of hardness and modulus to be determined.

INTRODUCTION

The microstructure of thin films plays a dominant role in determining the physical properties such as density and hardness and these properties in turn influence others such as the optical refractive index¹. The degree of porosity in deposited thin films may be controlled by bombarding the growing film with energetic particles. The refractive index of most dielectric films is both increased and stabilised through bombardment induced densification mechanisms. The most common method of achieving film densification is ion-assisted deposition (IAD) in which material is evaporated by electron beam bombardment and the depositing film is irradiated with low energy ions, usually O_2^+ in the case of oxide deposition.

Although there have been many studies of the influence of IAD on the optical properties of dielectric films there have been few investigations of the correlation in optical and mechanical properties. Here we report on the preparation of thin films of Ta_2O_5 and the effects of IAD on the optical refractive index, film density and microhardness.

EXPERIMENTAL METHOD

The experimental arrangement for film deposition is shown in Fig.1. The films were deposited in a cryo-pumped vacuum chamber by electron beam evaporation of high purity material (Superior Evaporants Inc, 99.9% pure). The base vacuum of the system was 1×10^{-4} Pa. The deposition rate was fixed at 0.5 nm s^{-1} . The system was also equipped with two ion sources. The first source was a Commonwealth gridless ion gun operated at an anode potential of 160 V (equivalent to a mean energy of approximately 100 eV O_2^+ for oxygen operation) and the second was a 2.5 cm Kaufman ion gun fitted with dual diverging grids that

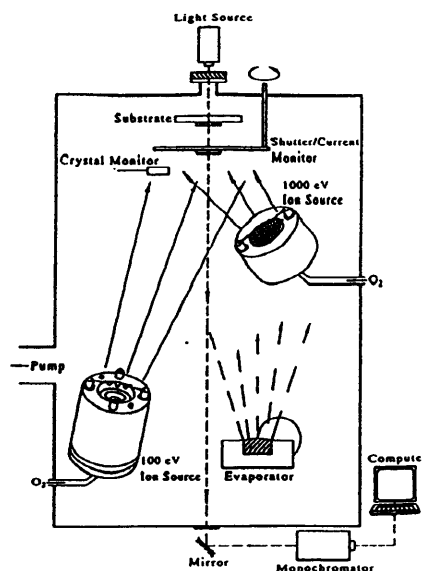


Figure 1. Schematic of the IAD thin film deposition facility

was operated at 1000 eV. Both ion sources were mounted at approximately 45° to the substrate normal and the evaporation source-substrate distance was 40 cm. The ion sources were operated with ultra-high purity oxygen gas and the ion current density at the substrate was varied from 0-200 $\mu\text{A cm}^{-2}$. The system was equipped with a quartz crystal rate monitor and an *in-situ* optical monitor for transmittance measurements at a wavelength of 633 nm during growth. The substrates used were glass microslides and semiconductor grade silicon wafers.

The deposited films were characterised for refractive index, hardness and density. The refractive index was calculated from the turning points in the transmission monitoring trace. Accurate measurements of the extinction coefficient of selected films were made using an absolute reflectometer. The film thickness was determined from the optical transmission measurements and also by Dektak profilometer measurements. In general the films were deposited to a thickness of approximately 1 μm . The film hardness was measured using a UMIS 2000 ultra micro-indentation system and measurements were made on both the glass and silicon substrates. The changes in density of the deposited films were assessed using Rutherford Backscattering Spectroscopy (RBS)².

RESULTS

Optical properties and density

The optical properties and density of the Ta_2O_5 films were found to be dependent upon the ion current density as shown in Fig. 2. The refractive index varied from 1.93 for the evaporated films to a maximum of 2.14 for the IAD films. The variation in the behaviour between the two ion energies (100 eV and 1000 eV) is related to the number of surface atoms that are activated by the incident ion and the probability of incorporation of excess oxygen in the film, both of which are energy dependent processes. The density was determined by RBS measurements. The density of the evaporated film (no ion assistance) was found to

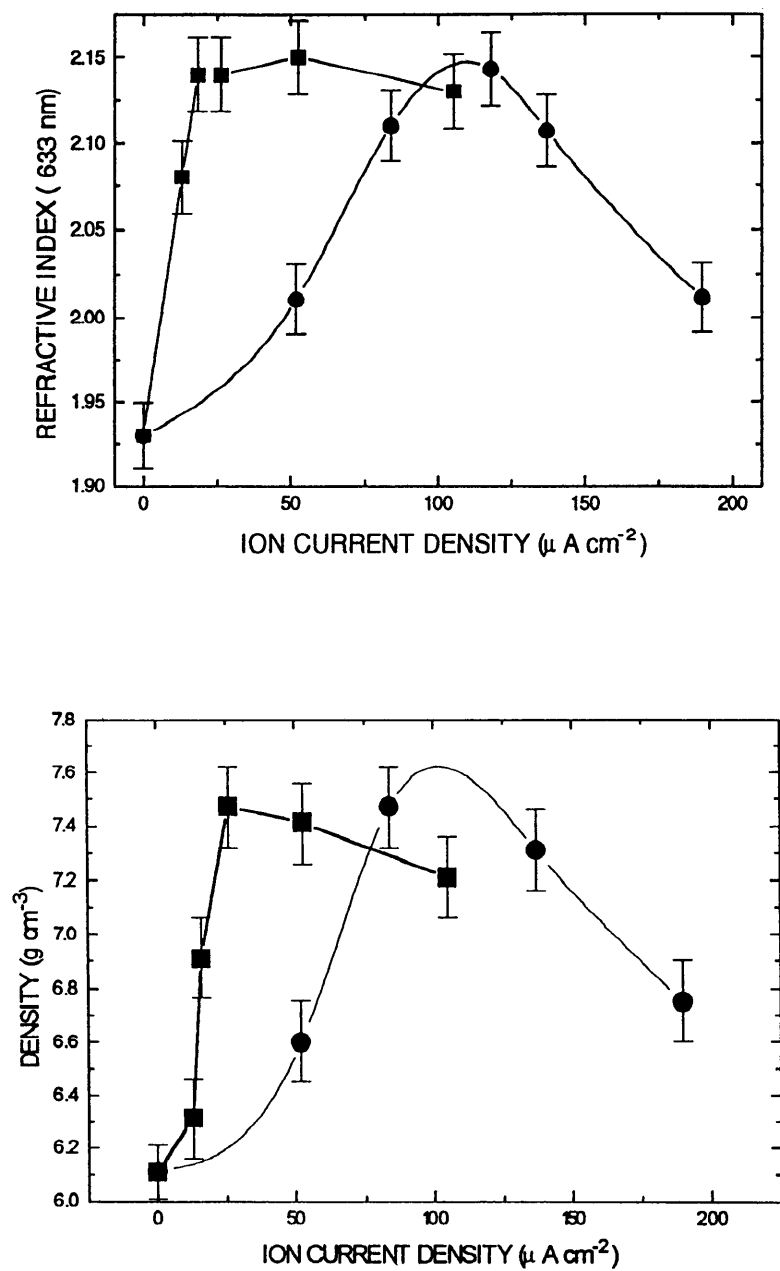


Figure 2. Optical refractive index (upper) and film density (lower) as a function of ion current density. ■ 1000 eV, ● 100 eV O_2^+ IAD

be 74% of the bulk density, and the maximum density of the IAD films (1000 eV ions) was found to be 93% of the maximum theoretical density. The maximum density for the 100 eV and 1000 eV IAD films occurs at an ion current density of 26 $\mu\text{A cm}^{-2}$ and 100 $\mu\text{A cm}^{-2}$ respectively.

Ultra-microhardness results

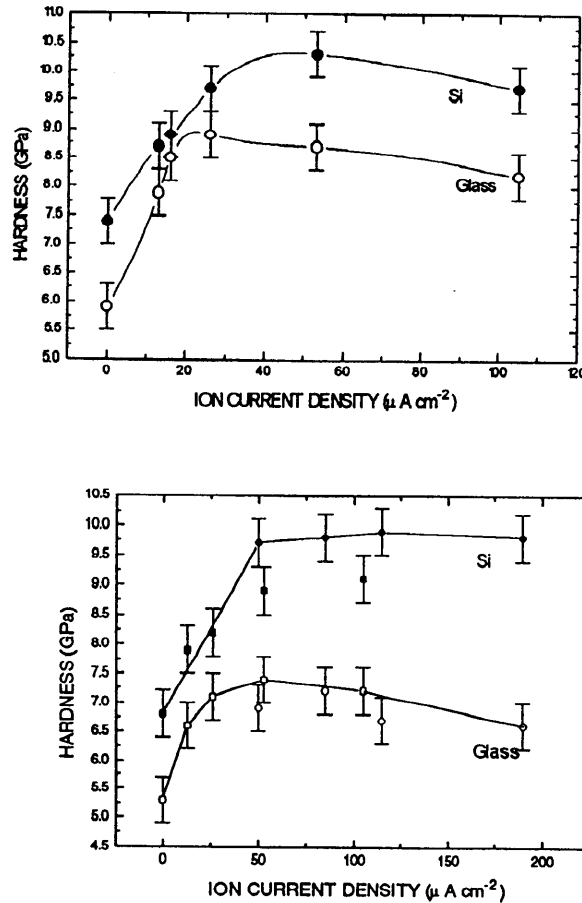


Figure 3. Hardness vs ion current density for films deposited onto Si and glass using 1000 eV IAD. Upper 3 mN loading, lower 7 mN loading

Figure 3 shows the hardness of films deposited on silicon and glass substrates when indented with a Berkovich diamond indenter. The depth of penetration was between 10%-20% of the thickness of the film. There are three major aspects of such observations: (1) the evaporated films show greater depth of penetration than the IAD film, (2) the extent of recovery for the evaporated films on glass is greater than the IAD films and (3) the evaporated and IAD films on the Si substrates show that the depth of penetration and residual impression depth is greater for the evaporated films.

When the films deposited onto Si were indented with a spherical indenter (5 μm radius diamond) significant cracking of the film was observed. However, for films deposited on glass substrates cracking occurred only in the case of the evaporated films. The critical load for cracking of the evaporated films was about 150-160 mN for both glass and silicon substrates, whereas for IAD films on glass the load was 330-340 mN.

The hardness or mean indentation pressure was determined using a novel load partial-unload technique³. Each data point was averaged over seven indentations. The variation in hardness

with bombarding ion current density is given in Fig. 4. No significant difference was found for films prepared with 100 eV or 1000 eV ions. The evaporated films were found to have lower hardness values than the IAD films and the hardness values for Ta₂O₅ films deposited onto glass always appeared lower than those deposited onto silicon.

Hardness and modulus as a function of depth

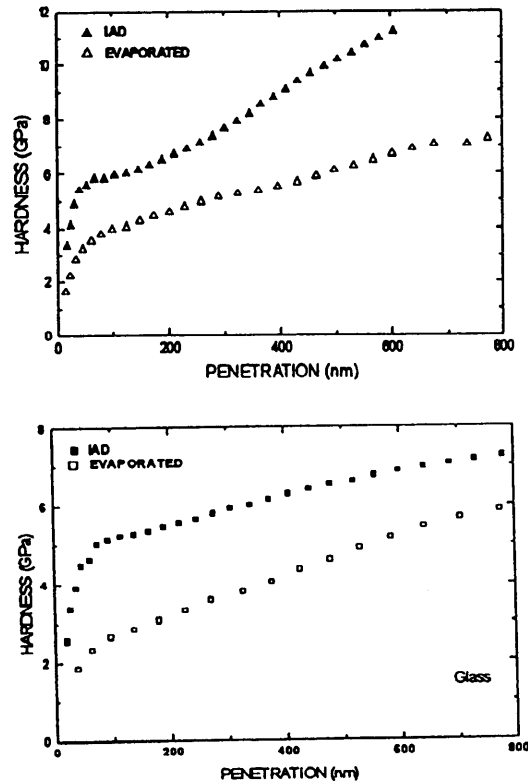


Figure 4. Hardness as a function of penetration. Upper Si substrate, lower glass substrate. Solid symbols IAD, open symbols evaporated only.

The analysis of the alternating load-partial unload data obtained with a spherical indenter enables the measurement of hardness and modulus with depth. The data in Fig.4 shows that initially the hardness rises reflecting the elastic response of the "blunt" spherical tipped indenter. At heavier loads, where the divergence of the data occurs, the behaviour is more controlled by the elastic-plastic response of the material. Comparative values of the hardness of the evaporated and IAD films on Si indicate that at 30 mN load the hardnesses plateau at 4.6 and 6.1 GPa respectively. In both cases, there is a well defined elastic region that is more pronounced for the IAD films. The hardness was found to rise rapidly for penetrations greater than 200 nm. This situation reflects the influence of the harder substrate

The variation of the elastic modulus with depth for the films deposited onto Si and glass substrates is shown in Fig. 5. For 30 mN loads the modulus of both evaporated and IAD films on Si is constant whereas the modulus decreases with depth for films deposited onto glass. For loads of 200 mN and greater depths of penetration the modulus reaches values comparable to that of glass.

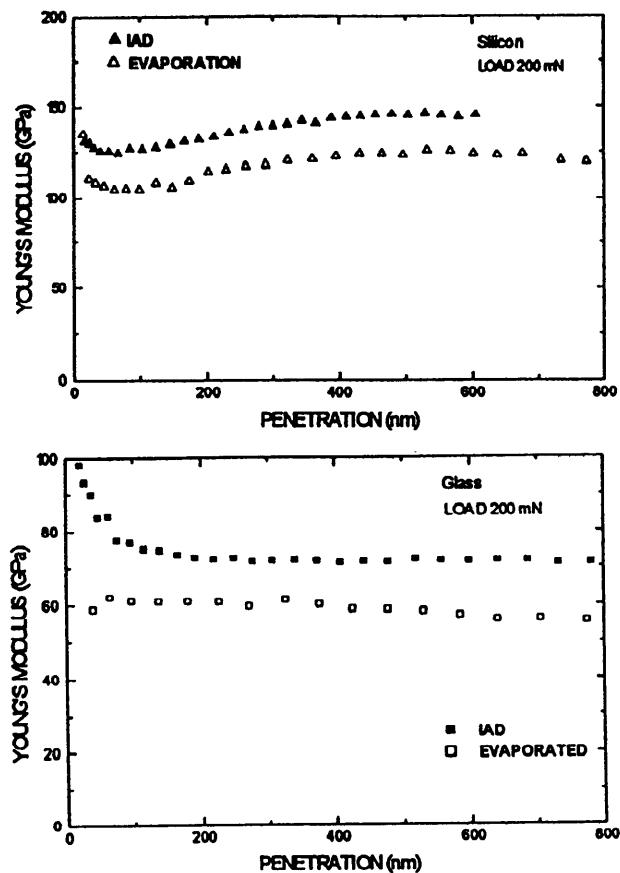


Figure 5. Elastic modulus as a function of penetration depth for 200 mN loading. Upper Si substrate, lower glass substrate

SUMMARY

Oxygen ion-assisted deposition of Ta_2O_5 films results in a densification of the growing film and an increase in the refractive index. The hardness of the deposited films increases with the degree of ion assistance and although no significant dependence of hardness on ion energy was detected the films deposited onto Si appeared to be harder than those deposited onto glass substrates even though the film thickness was of the order of $1\mu m$. The results indicate that for Ta_2O_5 films the substrate is influential on ultra-microindentation measurements of hardness and elastic modulus.

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Thin Films: Stresses and Mechanical Properties IV

Symposium held April 12-16, 1993, San Francisco, California, U.S.A.

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TA 418.9 T45 T463 1993



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