

Characterisation of ZrO₂ Thin Films on Glass Substrates

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Zirconia films are good material for use in many applications such as optical filters, laser mirrors, thermal barrier coatings, and gate dielectrics in microelectronic devices.

The films possess high heat resistance with low thermal conductivity, and have a high dielectric constant and refractive index. They are very transparent in the visible and near-IR region of the spectrum. Zirconia films are also chemically inert and have a high laser damage threshold.

Many methods have been reported for the production of zirconia films including sol-gel spin coating, atomic layer deposition (ALD), reactive sputtering and pulsed laser deposition. For the samples described here the ZrO₂ films were prepared by the electron beam evaporation of tablets of ZrO₂ in a reactive oxygen atmosphere.

For all applications of zirconia films it is necessary to have an accurate measurement of their thickness and refractive index, especially when the films are used as the high refractive index material in high-low index stacks for antireflection coatings or optical filters.

Spectroscopic Ellipsometry (SE) is an ideal technique for this task as it is non-destructive and able to extract both the film thickness and optical constants (refractive index and extinction coefficient) from the observed data simultaneously.

Sample Preparation

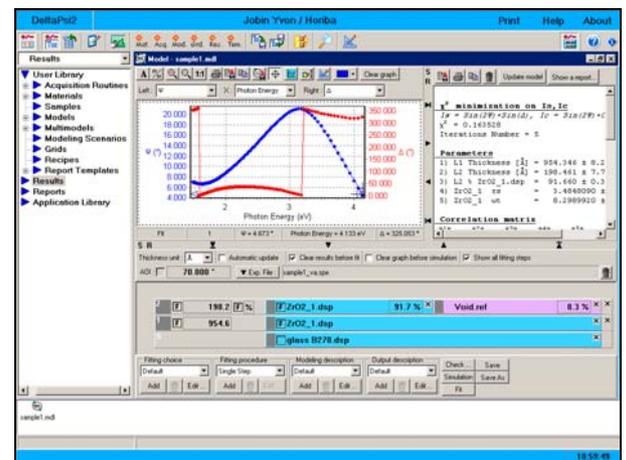
ZrO₂ films were prepared by electron beam evaporation of tablets of ZrO₂ in a reactive oxygen atmosphere. The glass substrates were 5 mm thick. The substrates were rotated during deposition to improve the uniformity of the deposited films.

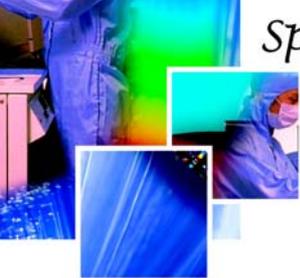
Results

The work was performed using a Jobin Yvon UVISEL Visible Spectroscopic Phase Modulated Ellipsometer. The Ellipsometric measurements were made at an angle of incidence of 70° across the spectral range 300-830 nm. Both the refractive indexes and thicknesses were extracted simultaneously from the SE data analysis.

The analysis were carried out in the transparent region of the material, and the Lorentz Oscillator formula was used to determine its optical constants. This formula minimises the number of parameters to calculate. If the analysis is extended to the semi-absorbing part of the spectrum then a Tauc-Lorentz oscillator model may be preferred.

When compared with RAE / RPE ellipsometers the phase modulated ellipsometer configuration is unique in that it allows high accuracy determination of the Δ angle across the full $[0^\circ, 360^\circ]$ range, even when Δ it is close to 0° . The reason for this is that Δ is determined from $\tan\Delta$ whereas the RAE / RPE ellipsometer allows only the determination of $\cos\Delta$.

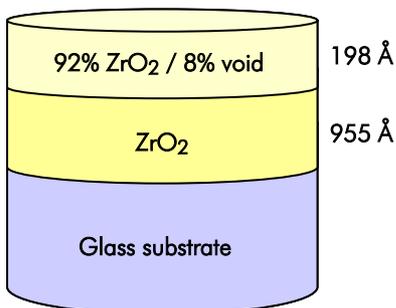




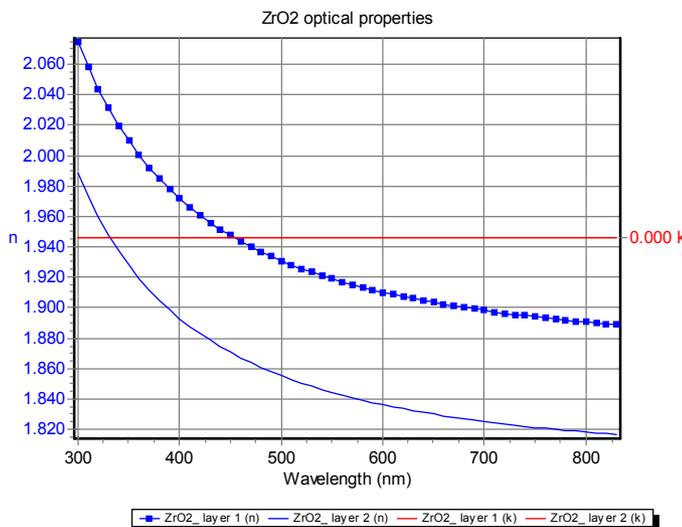
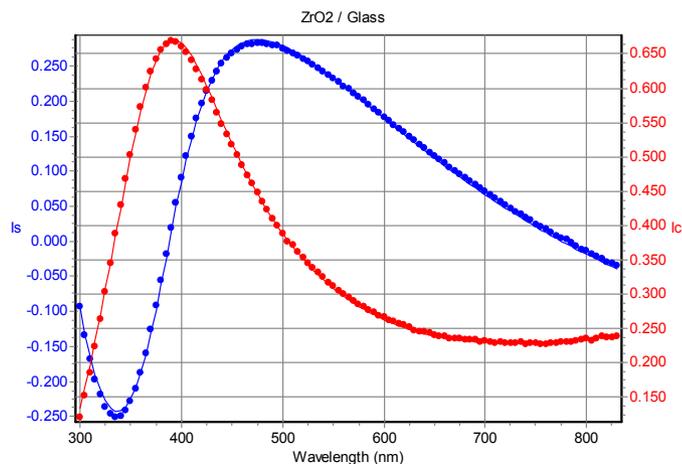
Characterisation of Zirconium Dioxide on Glass

Films directly evaporated from the ZrO_2 target can be described accurately using a two-layer model with a higher refractive index at the bottom and a lower refractive index on the top. The structure employed for the characterisation of the zirconia film is shown below.

The best model was found to have the top layer described by a mixture of 92% ZrO_2 and 8% void calculated using the Effective Medium Approximation. The experimental data and fitted curve are shown in the figure on the right. This behaviour was expected as non-stabilised zirconia films, such as prepared here, have a tendency to exhibit surface roughness. For applications where better surface roughness is necessary, so that there is less optical scatter, the films are modified by the addition of a stabilising materials such as Y_2O_3 , SiO_2 , MgO , CaO or CeO_2 .



The optical properties calculated for the ZrO_2 film are shown opposite. A value of $k=0$ shows that the film is transparent across the wavelength range studied.



Conclusion

The deposition method and conditions used for preparation of the ZrO_2 films have resulted in generation of an inhomogeneous porous layer. Owing to the sensitivity of the UVISSEL Spectroscopic Phase Modulated Ellipsometer and software it is possible to detect this layer, and to characterise its composition while at the same time measuring the film thicknesses and optical constants.

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