Deposition Techniques for High End Optical Coatings

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INTRODUCTION

From the simple broadband antireflection "AR" coatings used on our spectacles and camera lenses to the complex AR coatings used in military laser and optical fibre devices, optical thin films are to be found everywhere in our world. High quality coloured events and architectural lighting and night vision goggles both rely on optical bandpass filters technology whilst mirrors which selectively reflect wavelength allow "cool" lighting in our houses and operating theatres or efficient industrial UV curing processes. Although we may not be aware of their presence, thin films enable efficient operation of optical devices which would simply not be possible otherwise and coating technology is therefore a key process step in production of optical components.

These different filter types are well understood and there are already a number of useful guides [1] and software packages available to the designer to enable optimization of the basic thin film stack design. However, in choosing a manufacturing technique, the optical manufacturing engineer will also need to consider substrate size, geometry and material construction and what conditions such as temperature and humidity will be seen by the optical component in service.

This paper will present a short overview of the principles of the main manufacturing techniques available today and presents new experimental results for the mid frequency AC sputtering technique in particular before going on to discuss the significance of these results in the context of choosing manufacturing techniques for real industrial applications.

OVERVIEW OF CURRENT TECHNIQUES

Today's commercial techniques can generally be divided into the techniques of evaporation or sputtering and variants thereof.

Conventional evaporation

The most commonly used and longest lived of all techniques, evaporation relies on heating and subsequent evaporation of material from a source, linear propagation of the flux through the vacuum chamber and condensation at the substrate to form a coherent film. Alternate evaporation from two or more sources filled with different materials along with control of source power and coating time allows the build up of the stacks according to the required design. Dosing of process gases such as oxygen or nitrogen enables reactive processes and the control of film stoichiometry. Substrates are typically heated during deposition to get coatings with the best adhesion and optical performance. A whole range of source designs are available commercially including simple boats, electron beam guns and effusion cells according to the composition of source material to be evaporated

A number of "assisted" evaporation techniques have also come into common use with the aim of improving film qualities possible using conventional techniques where the relatively low vapour particle energies of the coating flux (0.1 to 1eV) have low surface mobility leading to only moderate film densities (80-90% theoretical) and some limitations in optical and mechanical stabilities depending upon the application.

In "Ion assisted" evaporation processes (IAD) a second ion source is used to produce inert and reactive gas ions which are accelerated and bombard the surface of the substrate during the deposition process. Momentum transferred to the surface atoms improves mobility and the voids and shadowing effects typical in conventional evaporation are reduced to produce a layer density closer to theoretical. In further enhanced so called "Plasma Ion Assisted" and "Ion Plating" variants, the evaporant material itself is also partly ionized and arrives at the substrate with much higher kinetic energies of the order of 10 eV allowing the production of much higher density films, quite often close to theoretical values with virtually no water absorption on exposure to the atmosphere. Such films are reported to exhibit higher refractive indices, improved adhesion and mechanical properties and stability with much reduced shift in their optical spectra when exposed to heat or humidity justifying the higher manufacturing costs relative to conventional evaporation.

Sputtering

Reactive magnetron sputtering is the mostly commonly used variant where a so called "target" manufactured from the desired coating material acts as the source and is held with the substrates in a vacuum chamber. Plasma of positively charged inert gas ions is formed close the surface of a "target". Strong magnetic and electric fields generated close to the target surface trap electrons to increases the number of collisions with inert gas atoms increasing the ionization rate. The charged

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ions are accelerated towards the surface of the target. Material from the source or "target" is then dislodged and ejected not by heating as in an evaporation process but by physical bombardment and momentum exchange from the impact of energetic particle impact. Target material is emitted mostly as neutral atoms which are unaffected by the magnetic field, transported through the system and condense at the substrate which is in relatively close proximity. Magnetron sputtering was first developed in the 1970s but is now beginning to find more widespread use in the production of optical coatings as sophisticated power supply and process control technologies enable better control of the reactive processes usually necessary for optical layers. It is also reported that the high kinetic energies and mobilities associated with the process can also enable the formation of dense, stable films with outstanding mechanical and optical stabilities when exposed to high temperatures and humidities .

Ion Beam Sputtering (IBS) is a high energy coating process where material is sputtered from the target by an ion beam generated by a separate ion source. This technique is characterized by high energy coating flux at around 20 eV and has become established for manufacture of coatings with amongst the best mechanical and optical stabilities albeit with low throughput.

Different evaporation and sputter techniques and their reported benefits are discussed in detail by Tiziani, Hentschel and Braunecker [2] and further reading sources referenced.

Whatever the technique, a better understanding the relationship between properties and film structure is key in using and developing all these techniques further. Recent "round robin" experimental studies [3] have made direct comparisons between single layers of oxides of silicon, hafnium, tantalum and niobium deposited by a range of evaporation techniques, RF magnetron and ion beam sputtering . Strong correlations between refractive index, thermal shift and mechanical stress were reported with pore fraction and type as the main controlling microstructural feature and the authors concluded that a certain pore volume was essential in achieving optimum films with balanced mechanical and optical properties.

Experimental data is now presented on optical stabilities achieved for a different sputtering process , mid frequency AC magnetron sputtering and pulsed DC, and examines process repeatability in particular.

EXPERIMENTAL SET UP

Coating were produced in a vertical batch sputter system (Figure 1) , batch size 40 pieces of substrate 180x136 mm, equipped with turbomolecular and Meissner pumping capability. Four vertical sputter cathodes 635 x 125 mm were configured for reactive sputtering in mid frequency AC and pulsed DC operation. Substrates were attached to central rotating drum of 1m diameter. No uniformity masks or shapers were necessary between cathode and substrates due to a proprietary cathode tuning system Film chemistry was managed with closed loop plasma emission monitoring for control of argon and oxygen gas flows during sputtering. Broadband optical monitoring with direct substrate measurement was used for end point termination.

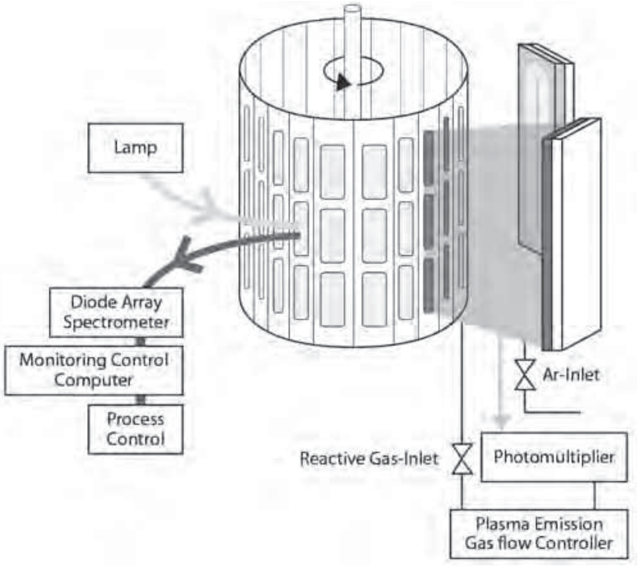


Figure 1: Schematic of vertical sputter system with broadband optical and plasma emission monitoring.

Two different filter types were manufactured:

Type 1:	UV/AR Filter
Function:	A combined UV Blocking and Broadband antireflection coating for the visible Materials: niobium oxide (Nb_2O_5) and silicon dioxide (SiO_2)
Substrate:	Borosilicate glass 180x136mm
Stack design:	Side 1 AR, 4 layers Side 2, UV blocking 28 layers

Type 2:	Fabry Perot Filter
Function:	Bandpass filter with centre wavelength (CWL) 550 nm and full width half maximum (FWHM) 35.6 nm
Materials:	Niobium oxide (Nb_2O_5) and silicon dioxide (SiO_2)
Substrate:	Borosilicate glass 180x136 mm
Stack Design:	Side 1, 35 layers

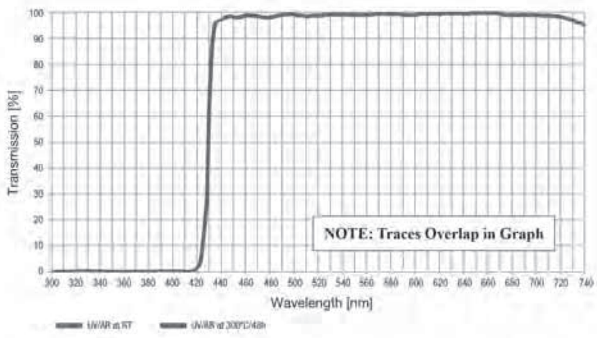
Nb_2O_5 and SiO_2 sputter rates were 0.4 nm/s and 0.35 nm/s respectively for both processes.

Temperature and environmental stability were tested for the combined type 1, UV/AR blocking filter using three samples from the batch for each test. Optical spectra were recorded at room temperature immediately after deposition and again after extended high temperature exposure at 300°C for 48 hours. Environmental tests were carried out according to MIL-C-14806A [4] which includes individual test elements for humidity, salt water fog and hardness. Optical spectra were recorded before and afterwards for comparison.

Process repeatability was tested using the type two, Fabry Perot filter. The process was repeated six times over a 30 day period on four different days. Batches 1 and 2 were produced consecutively on day 1, whilst batches 5 and 6 were produced consecutively on day 30. Batches 3 and 4 were produced deliberately at intermediate times on non-consecutive dates following coating work using other processes to check the influence on process results. Following initial set up of batch 1, no further process set up trials were carried out .Batches 2, 3, 4, 5 and 6 were executed without any test runs . Measurement samples were taken from the centre point of the subtstrate holder for each batch

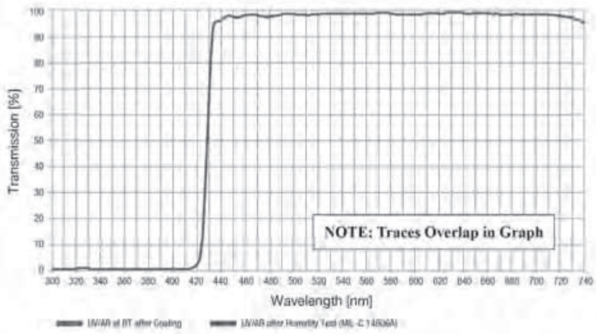
SUMMARY OF EXPERIMENTAL RESULTS

Temperature and environmental stability test results for the UV/AR filter are shown in Figures 2 and 3 respectively. Base spectra of transmission versus wavelength measured on the both samples immediately after coating is well within the coating specification which is representative of a typical specification in mass production. Following the high temperature test we see very small shift of 0.1% in absolute transmission in the blocking range, which is within the range of measurement tolerances. A shift of 0.3% average transmission over the visible range shows a small amount of remaining absorption but the T_{50} position remains at 428.8 nm. Similarly in the environmental tests, we see a 0.1 nm shift in the T_{50} position and a 0.1% shift in absolute transmission over the visible with no shift of performance in the UV.



Substrate	T < 1% abs. 300 - 405nm (%)	T = 50% at 430 ±3.5nm (nm)	T > 96% avg. 440 - 720nm (%)	T > 94% abs. 450 - 720nm (%)
UV/AR at RT	0.29	428.8	99.0	97.9
UV/AR 300°C/48h	0.30	428.8	99.3	98.3

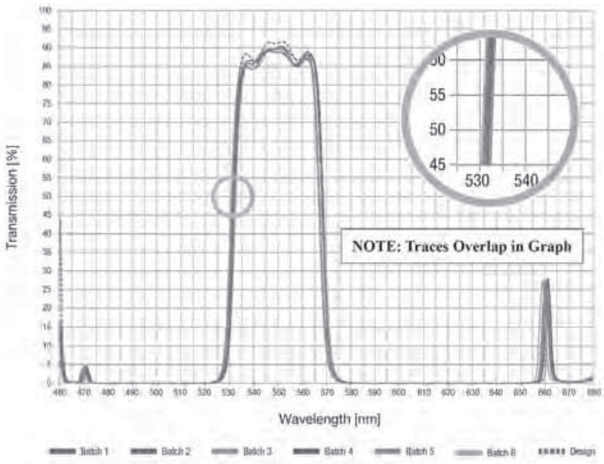
Figure 2: Optical transmission spectra for UV/AR filter at room temperature prior and post high temperature soak test.



Substrate	T < 1% abs. 300 - 405nm (%)	T = 50% at 430 ±3.5nm (nm)	T > 96% avg. 440 - 720nm (%)	T > 94% abs. 450 - 720nm (%)
UV/AR at RT After Coating	0.31	428.5	99.0	97.7
UV/AR after Humidity Test (MIL-C 14806A)	0.31	428.6	99.0	97.8

Figure 3: Optical transmission spectra for UV/AR filter at room temperature prior and post environmental test according to MIL-C-14806A.

T_{50} positions of both rising edge and falling edge for the Fabry Perot filter across all six batches are illustrated in Figure 4. Overall repeatability is better than 1.79 nm (+/- 0.17%) and 2.03 nm (+/- 0.18%) for the rising and falling edges respectively. Data for consecutive batches such as 1 and 2, or 4 and 5 shows much smaller shifts of 0.2 nm or less in the T_{50} positions. For comparison, in a conventional hot evaporation process we would expect humidty shifts of greater than 1% and shifts greater than 1%, and 3% for temperature tests at 120 and 300°C respectively.



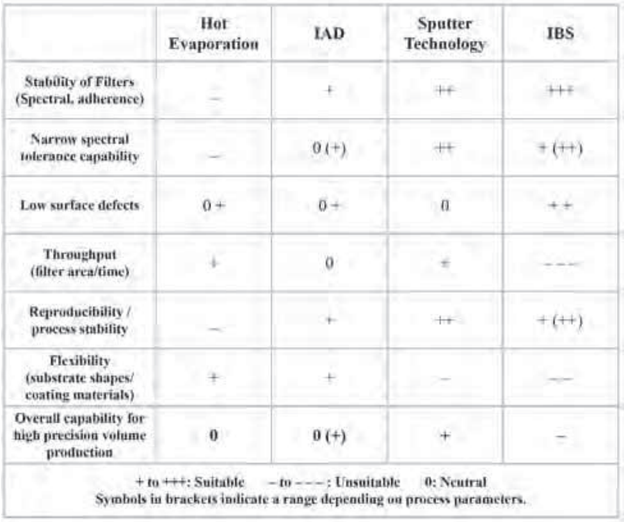
	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6	Design	max-min (nm)	max-min (%)
T=50% (rising edge)	531.04	531.19	532.45	531.84	530.86	530.66	531.71	1.79	0.34
T=50% (falling edge)	566.89	566.91	568.53	567.88	566.81	566.50	568.07	2.03	0.36

Figure 4: Optical transmission spectra to show repeatability of Fabry Perot process.

These results could have significant consequences for the optical manufacturing engineer and his decision for choice of thin film deposition process.

The sputtered UV/AR films demonstrate excellent optical stabilities in environmental and high temperature soak tests suggesting the deposition of dense layers and confirming that the basic technique is well suited to high precision processes demanding stable films. Even more significantly however, the narrow spectral precision achieved in this sputter configuration without test runs and in non-consecutive manufacturing batches further reinforces the stability of the technique and shows how test runs necessary in convention evaporation processes or other sputter configurations could be eliminated removing process set up times and costs. For the manufacturing engineer this opens up the possibility to run different processes "back to back" efficiently.

However, these are not the only factors to consider in determining what manufacturing technique. The low temperatures associated with the sputter process of 100^{o}C reinforce its suitability for temperature sensitive substrates. On the downside, the capital investment costs are usually higher than for evaporation and the sputtering process is restricted to common metals and oxides. With its ability to handle different substrate shapes and source materials including metals, oxides, fluorides and sulphides, conventional evaporation is a flexible technique. Relatively high temperature processing at temperatures up to 200°C means limitations for certain cemented or polymer substrate types but the initial capital investment costs can be low and the performance of the coatings is perfectly adequate for many applications. The more recent IAD technologies have higher investment but the ion sources required can sometimes be retrofitted to conventional systems and the increased running costs can be offset by the improved spectral precision and better manufacturing yields where optical specifications call for tighter tolerances. A summary of the relative advantages of different techniques is presented in Figure 5.



	Hot Evaporation	IAD	Sputter Technology	IBS
Stability of Filters (Spectral, adherence)	–	+	++	+++
Narrow spectral tolerance capability	–	0 (+)	++	+ (++)
Low surface defects	0 +	0 +	0	+ +
Throughput (filter area/time)	+	0	+	– – –
Reproducibility / process stability	–	+	++	+ (++)
Flexibility (substrate shapes/ coating materials)	+	+	–	– –
Overall capability for high precision volume production	0	0 (+)	+	–

+ to +++: Suitable – to – – –: Unsuitable 0: Neutral
Symbols in brackets indicate a range depending on process parameters.

Figure 5: Comparison table for thin film deposition techniques.

CONCLUSIONS

The experimental data on environmental tests presented for sputtering shows the ability of the technique to produce highly stable layers with high optical precision.

The repeatability tests in the particular configuration further show the potential to run different processes back to back eliminating the need to have set up or test runs prior to changing manufacturing process.

No single technique is the ideal solution for all optical coating challenges. The inherent flexibility of evaporation based processes with simple change of substrate types and coating materials means they looks set to remain a mainstream manufacturing process for the foreseeable future. However, sputtering techniques such as mid frequency AC / pulsed DC can have significant advantages as mass production tool for high quality films.

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