



Chemical Vapour Deposition of High Precision Optical Coatings

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ABSTRACT

In this paper we describe how optical coatings can be deposited uniformly with a high precision and reproducibility on 3-dimensional substrates, such as spherical lenses, by CVD. We present results that will highlight some specific advantages of CVD over the traditionally used methods of e-beam evaporation and magnetron sputtering and we will show that CVD has tremendous potential for enhancing the quality of optical coatings and for making cost savings.

1 INTRODUCTION

Requirements for coated precision optics components are changing rapidly, driven by the expanding growth in markets such as optical telecoms, data storage and displays. In addition there is a general trend in these markets towards miniaturisation of systems, leading to reduced size and higher functionality and performance of components and sub-systems. These needs are pushing conventional technologies based on physical vapour deposition (PVD) to higher levels of control and throughput, but there are limitations arising from the tooling required for the line-of-sight PVD methods. Moreover, use of PVD to accommodate micro-optical components and to achieve the necessary precision and uniformity across 3D micro-optic surfaces is highly problematic. Commercial demands on optical coating production are also driving the need for higher throughput processes for applications such as flat panel displays, liquid crystal display (LCD) projection systems and visible/thermal control coatings for low cost imaging systems.

To meet all these demands, coupled with the additional need for optical thickness precision control to fulfil the increasing criteria for optical coating spectral performance, requires production implementation of high throughput precision processes with uniformity control over complex areas and good reproducibility both within a batch and on a batch-to-batch basis.

Chemical vapour deposition (CVD) offers a non-line of sight deposition method well suited to economical coating of 3D micro-optical componentry. In CVD a thin solid film on a surface is synthesised from the gaseous phase as a result of chemical reactions occurring at the gas/surface interface [1]. It is this reactive process which distinguishes CVD from PVD processes. The prime benefit of CVD arises from the fact that the deposition process occurs by a molecular reaction on the substrate surface and this means that it is possible to coat intricate solid shapes, such as spherical and cylindrical lenses, with layers that are of uniform thickness and composition. This is in contrast with PVD processes where the deposition is line-of-sight, which precludes the possibility of coating 3-D objects uniformly without complex movement of either the substrate and/or the deposition source.

Another outcome of the non line-of-sight deposition by CVD is that it is also possible to simultaneously coat opposite substrate surfaces so providing enhancement in product throughput. In addition, because the film is built up in a molecular fashion on the substrate surface a CVD deposited layer is usually denser with a higher refractive index, smoother morphology and better crystalline quality than can be achieved by PVD processes. A further advantage of CVD over PVD follows on from the 'all round coating' capability in that a large number of a substrate samples can be coated simultaneously, so the high throughput requirements increasingly needed for precision optics applications can be more readily met by CVD than by PVD. Furthermore, in a CVD reactor the environment is generally cleaner with less contamination by dust than in a PVD reactor, since liquid precursors are used as opposed to solid material evaporation sources, and this allows better control of product cosmetic quality.

Because CVD involves chemistry there is the benefit of a wider range of materials and compounds that can be deposited by CVD than PVD [1]. This is particularly advantageous for optical coatings which, depending on the application and operating region of the spectrum, can consist of a single layer or a hundred or more layers of a

range of materials. For example, for low refractive index materials silicon dioxide can be used while for high refractive index cases the oxides of zirconium, titanium, niobium, hafnium or tantalum may be employed. And for transparent conducting layers, materials such as indium tin oxide are of interest. CVD is ideally suited to the preparation of such varied layers. A further distinctive feature of CVD is that it allows for deposition of novel materials that would offer significant design/performance benefits over standard layers and which would be extremely difficult to produce using PVD technologies.

A comparison of CVD and PVD techniques for optical coatings is summarised in Table 1.

Table 1 Summary of Features of CVD & PVD

Characteristic	PVD	CVD
Principle	Physical processes	Molecular reaction
Range of materials	Limited	Very extensive
3-D capability	Very difficult	Relatively easy
Sample throughput	Low	Extremely high
Morphology/ Crystallinity	Limited control	Very controllable
Novel Materials	Very difficult	Relatively easy
Sample contamination	Can be high	Very low
Cost per sample	X	< 10% of X

In this paper we illustrate some of the advantages mentioned above for CVD with results for single optical coatings of tantala and silica, as well as multilayers.

2 EXPERIMENTAL

The equipment used for the CVD of tantala, silica and multilayers was a standard low pressure CVD (LPCVD) system [2]. Tantala films were grown from tantalum pentaethoxide and silica layers were deposited using diacetoxydibutoxysilane. The liquid precursors were contained in specially designed, thermostatically controlled bubblers that allowed a constant and reproducible pick up of the vapour by an inert carrier gas such as nitrogen. A gas handling system allowed the introduction of the precursors into the CVD reactor.

The substrates consisted of flat witness pieces of BK7 glass, fused silica and single crystal silicon and were positioned in the heated reaction zone of the reactor using proprietary designed holders. Deposition temperatures in the range 350-450°C and reactor pressures of 0.3-10 torr

were employed. The optical properties of the deposited layers were analysed by dispersive UV/vis and IR spectroscopy as well as by fixed wavelength (632.8 nm) ellipsometry. Layer thicknesses were determined by ellipsometry and surface profilometry. Surface quality was examined with AFM and environmental stability was evaluated with an accelerated lifetime test of exposure to saturated steam at 15 psi gauge for 24 hours. Abrasion resistance was assessed with the red rubber test (MIL C675C).

3 RESULTS AND DISCUSSION

Figure 1 shows the variation of deposition thickness with time for silica. Such excellent linearity allows a wide range of layer thicknesses to be deposited for different optical coating designs. It also provides the precision and accuracy that are required for high quality optical thin films.

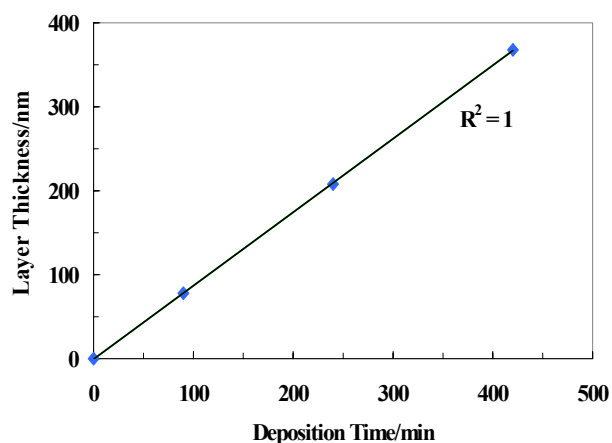


Figure 1 Variation of silica growth rate with time

The non line-of-sight feature of CVD is illustrated by the results in Figure 2 which shows the spectral characteristics for thin tantala films on the opposite sides of pairs of glass plates held together so that deposition could only occur on one face of each plate. After deposition the plates were separated and the spectra were then recorded for the single layer deposits. There is remarkable reproducibility for the films on both sides of the pairs of plates for all of the samples.

As discussed above, CVD layers are generally denser than those prepared by PVD and such dense structures will provide drift free optical properties close to those of bulk material. The high optical quality achievable is illustrated in Figure 3 for CVD tantala which has an exceptionally low absorption over a wide wavelength range.

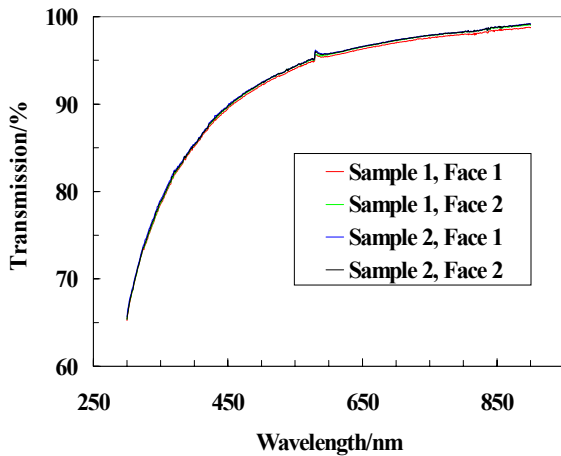


Figure 2 Transmission spectra for thin, single layers of CVD tantalum grown on opposite faces of pairs of glass plates

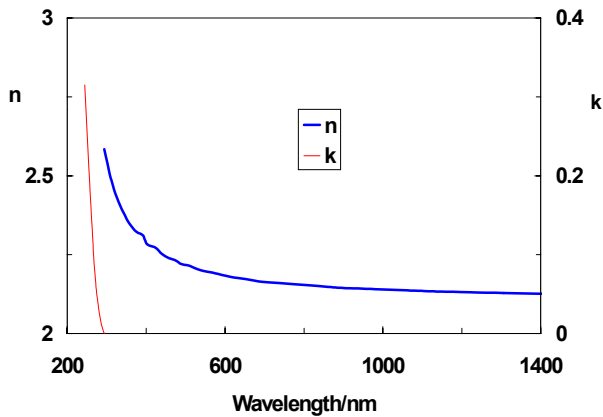
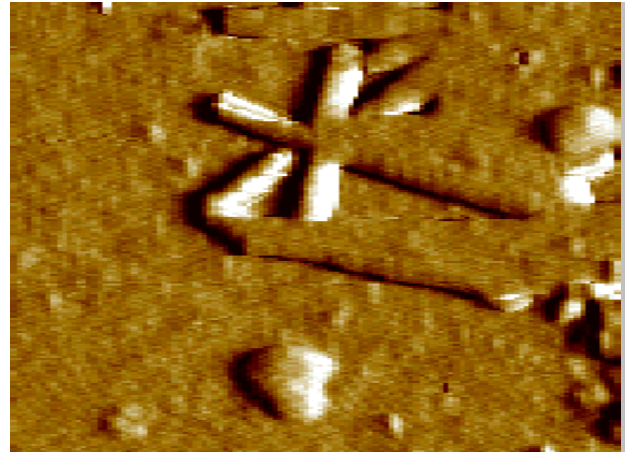


Figure 3 Variation with wavelength of refractive index and absorption coefficient for CVD grown tantalum

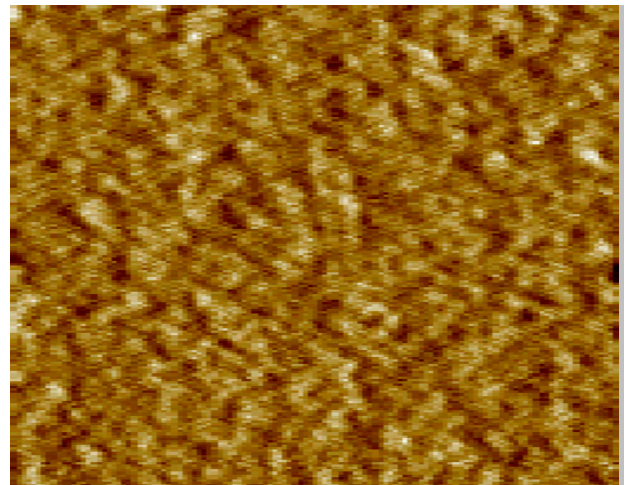
As also mentioned above, an important feature of CVD processes is the better control of the cosmetic surface quality compared with PVD. This is demonstrated by the comparison of AFMs for a CVD and PVD layer of the same oxide - Figure 4. Both samples have rms roughnesses of ~ 0.3 nm, but the better surface appearance of the CVD layer is evident. The amorphous, dense film structures that can be prepared by CVD will give low scattering capabilities for demanding optical applications.

The mechanical robustness of CVD layers is shown both by an accelerated lifetime test and an abrasion test. Oxide layers kept in saturated steam at 15 psi gauge for 24 hours showed no sign of delamination. And after 50 strokes of

the red rubber test there was no visible evidence of marking on the surfaces of layers. Clearly, CVD films have the potential for applications in extreme environments and without the need to take any special handling precautions.



(a)



(b)

Figure 4 Comparison of surface morphology of oxide coatings grown by (a) PVD and (b) CVD

Although results have been presented here only for two types of optical thin films, the same approach can be used for other optical coatings such as titania, alternative high refractive index oxides, and fluorides.

For many designs of optical coatings a single thin film is inadequate to achieve the required optical characteristics and multilayers are needed. Figure 5 shows the transmission spectrum for such a coating designed to have $< 0.5\%$ reflectance at 1550 nm. The three layer coating

made up of silica and tantala evidently provides the desired feature. The spectrum in Figure 4 was measured for a flat witness piece with the coating deposited simultaneously on both sides of the substrate plate. This again illustrates the all round coating capability of CVD, particularly since there was no marking at all on any surface arising from the substrate holder. This is achievable through the non line-of-sight deposition achievable with CVD. This capability of coating 3-D objects uniformly all over is shown more clearly by reflectance spectra taken at five different positions on a 2 mm ball lens – Figure 6. The variation in the minimum reflectance is better than $\pm 0.25\%$ and there are no equatorial lines such as are commonly found with PVD techniques.

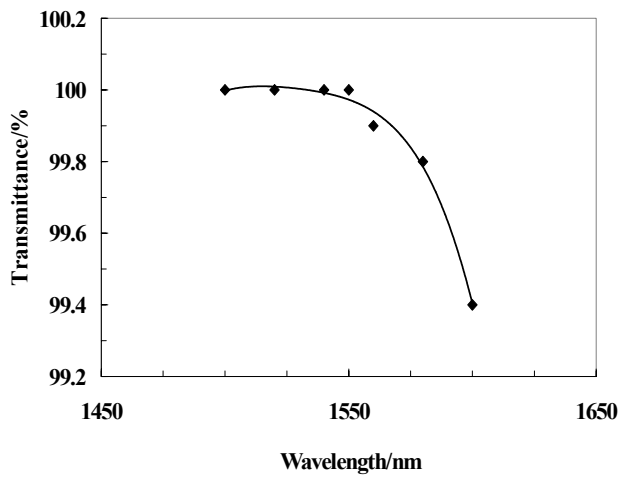


Figure 5 Transmission spectrum for a CVD AR coating at 1550 nm

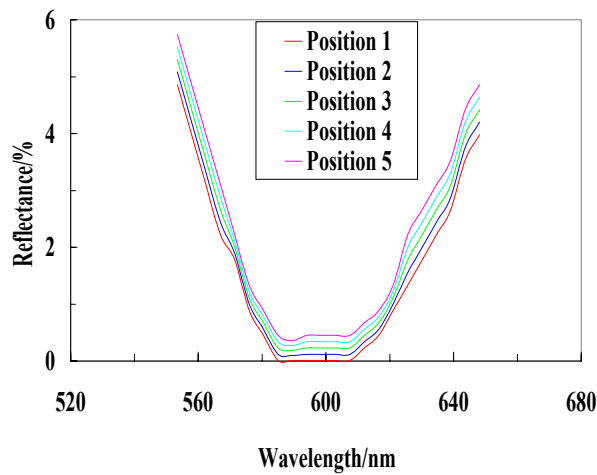


Figure 6 Reflectance spectra for a CVD coating at five different positions on a 2 mm ball lens

4 CONCLUSIONS

The results presented in this paper demonstrate the inherent advantages of CVD over PVD processes for the preparation of optical coatings from the point of view of materials quality and optical characteristics. By careful design of the tooling to hold the substrates it is also possible to simultaneously coat thousands of micro-optic samples, such as ball lenses, leading to low coating costs per sample. These features, together with the capability of CVD to deposit a wide variety of materials, indicates the significant potential that CVD has for the preparation of high precision and high quality optical coatings with demanding characteristics.

4 REFERENCES

- 1 M.L. Hitchman in *Chemical Vapor Deposition: Principles and Applications* (eds. M.L. Hitchman and K.F. Jensen), Ch. 1, Academic Press, San Diego, 1993
- 2 M.L. Hitchman, J. Kane and A.E. Widmer, *Thin Solid Films*, **59**, 231, 1979