Stability and Repeatability of 2-Layer Anti-Reflection Coatings

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ABSTRACT

The manufacture of a simple anti-reflection (AR) coating design can often prove to be more difficult than anticipated. The case described here required near zero reflectance (0.15%) at a single wavelength at two angles of incidence (0E & 30E), design criteria which can easily be met in theory with a simple 2-layer structure using materials such as ZrO_2/SiO_2. However, when manufacturing tolerances were brought into play, variations in film thickness and film index made it difficult to produce coatings which consistently met the requirement.

By changing to a different material system (MgF_2/TiO_2), we were able to use a different 2-layer design which is much less sensitive to these process variations and which improved the yield to 100%. The theoretical basis for this reduced sensitivity is given, together with confirming experimental results.

INTRODUCTION

It has been almost axiomatic in the field of optical coatings that the design stage of any coating exercise program is the easiest; attempting to implement the design in practice is where all the difficulties lie. In a sense this is true if one considers the design as a static illustration of what might be achieved, an ideal performance goal to be sought but not realized because of limitations in the manufacturing process. In recent years, modeling programs have become much more sophisticated and now tools such as *Reverse Engineering* and *Error Tolerance Analysis* are available to allow us to analyze manufacturing errors and predict their effect on yields. The design exercise has become much more of an iterative process whereby now, instead of thinking in terms of the design as perfect and the process as flawed, we have come to think of the design as deficient if it does not take account of inevitable manufacturing errors and still allow high yields.

In this paper, we describe a simple example of how important it is for the design and manufacturing process to be developed together. We recently had the opportunity to produce an AR coating on a large prism substrate made of SF11 glass for Kaiser Electro-Optics. The coating requirements are set out in Table I. Fortunately, we had a good working relationship with KEO because the task turned out to be rather more difficult than we anticipated.

The requirements in Table I, although demanding very low reflectance over a range of angles of incidence, only apply at a single wavelength, and so can be quite easily satisfied with a simple 2-layer structure of high and low index materials - the ‘V’ Coat. (Figure 1)

TABLE I: Coating Requirements for KEO Prisms

Substrate Index	1.785 (SF11)
Reflectance, Surface A	R < 0.2% @ 543nm
Reflectance, Surface B	R < 0.15% @ 543nm
Angle of Incidence	0 - 30 deg.
Durability	MIL-M-13508

The nature of the prism limited on process in 2 ways: it received other coatings and treatments, some before the AR, some after, and those treatments limited the process temperature to 100^{o}C. Also, the height of the prisms was such (~ 6”) that they hung “low” from their planets in the coating chamber, resulting in emission and deposition angles as high as 45^o at some points during the rotation. The temperature limit led us initially to select SiO_2/ZrO_2 as our coating materials because they are robust at low substrate temperature. The high emission angles undoubtedly contributed to the poor control which we subsequently found with the SiO_2/ZrO_2 system.

Our first attempt to produce satisfactory prism coatings was with Design 1, shown in Table 2. We soon found that the results with this design were quite inconsistent; if a layer thickness correction was made to improve the performance, it might result in a larger or smaller shift than was intended. If a good run was simply repeated, the next run might shift out of specification. Obviously, our level of control with this process and design was inadequate. To find a solution, we adopted the following analytical approach:

Phase I

- Perform 10 consecutive runs with Design 1 – no changes.
- Reverse Engineer the layer thicknesses and indices for each of the 10 runs.
- Use these results to estimate standard deviation (S.D.) of thickness errors.
- Perform an error tolerance analysis to predict process yield with Design 1, based on established S.D. errors.

We expected to find that the predicted yield with Design 1 would be unacceptably low, and this turned out to be true. We proceeded to Phase II.

Phase II

- Try a second Design 2, (TiO_2/Mg F_2) selected because it should be less sensitive to thickness variations.
- Using thickness errors from Design 1, predict process yield for Design 2 (Tolerance Analysis).
- Produce 10 runs with Design 2.
- Reverse Engineer thicknesses and indices – compare S.D. errors with those for Design 1 materials.
- Revise the yield prediction based on new thickness error values from Design 2 results.
- Evaluate durability of Design 2 coatings.
- If yield prediction is 100%, set up production.

Having worked through this procedure, we were indeed able to establish a process using Design 2 which has, to date, yielded 100% good prism coatings. However, the analysis did reveal some unexpected results: The decreased sensitivity of Design 2 is not enough in itself to make it viable if thickness errors are the same as for Design 1. What emerged as critical to the success of Design 2 was that the MgF_2 layers were much more repeatable than the equivalent SiO_2 layers in Design 1.

‘V’ COAT THEORY

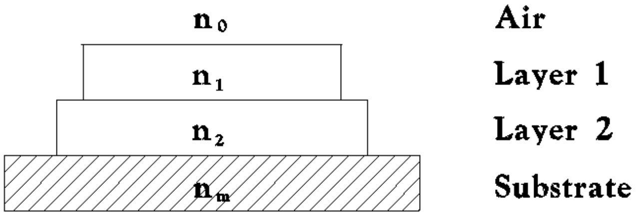

Figure 1: ‘V’ Coat Layer Structure

The performance of a 2-layer AR coating has been described and analyzed in much previous work[1]. The structure is shown in Figure 1. It can readily be shown that if the 2 layers each have λ/4 optical thickness, the reflectance will be zero at the design wavelength if the indices satisfy the requirement:

$$n_2^2 \bullet n_0 = n_1^2 \bullet n_m$$

If the indices are not perfectly matched it is still possible to achieve zero reflectance by adjusting the layer thicknesses. In this situation there are two solutions with thicknesses are given by[1]:

$$\tan^2 \delta_1 = \frac{(n_m - n_0)(n_2^2 - n_0 n_m) n_1^2}{(n_1^2 n_m - n_0 n_2^2)(n_0 n_m - n_1^2)}$$

$$\tan^2 \delta_2 = \frac{(n_m - n_0)(n_0 n_m - n_1^2) n_2^2}{(n_1^2 n_m - n_0 n_2^2)(n_2^2 - n_0 n_m)}$$

where δ_1 & δ_2 are the phase thicknesses of layer 1 and layer 2 respectively. Figure 2 shows δ_1 & δ_2 plotted against the refractive index (n_2) of layer 2, when the substrate has an index of 1.785 (n_m) and the layer 1 is silica (n_l=1.46). Two features of Figure 2 are pertinent to the discussion:

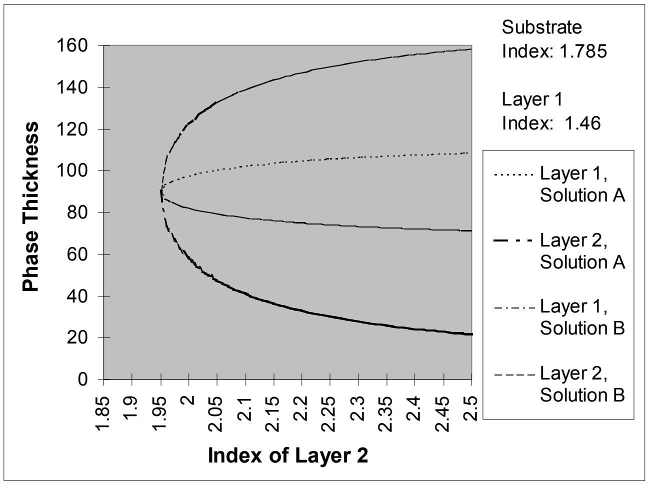

Figure 2: Thickness Solutions for Zero Reflectance

- There is no zero reflectance solution for $n_2 < 1.95$, i.e., to be successful with a silica top layer and an SF11 substrate, we must have a high index of at least 1.95.
- When n_2 is close to 1.95, the required thicknesses are very sensitive to a changes in n_2. If n_2 is greater, say 2.3 to 2.5, this sensitivity is much less.

THE ANALYTICAL METHOD

In each coating run there was one witness coupon of SF11 (n = 1.785) and one coupon of borosilicate glass (n = 1.52). The coated coupons were measured in reflectance over the visible wavelength region at both near normal (6°) and 30° angles of incidence. Thus, for each run we generated four reflectance spectra.

Reverse Engineering

Taking each measured spectrum as a set of optimization targets, we used our thin film design program - The Essential Macleod - to refine the layer thicknesses, and layer indices (in terms of packing density) until the theoretical performance matched the measured as closely as possible. The thicknesses and indices thus obtained were assumed to represent the real values for the run being analyzed. For consistency each refinement started from the same design, but after a solution had been found, it was perturbed a few times to ensure that the refinement would always return to the same answer.

We hoped, but did not expect, that the four spectra from each run would all result in the same reverse engineered design as this would have been a powerful validation of the procedure. In fact, we found that the results matched very well by substrate type: for each run, the spectra on glass gave essentially identical results at both angles of incidence. The SF11 spectra also gave similar results at both angles of incidence, but they were consistently different from the glass results. For this reason, the four original spectra were combined into two sets of optimization targets and two results were reverse engineered for each run: One for glass at both 6 ^{o} and 30^o; the other for SF11 at 6 ^{o} and 30^o.

We did not attempt to explain this discrepancy between the results for the two substrate types as it did not hamper our analysis - both showed the same pattern of thickness and index variations.

In this way we generated sets of thicknesses and indices for 10 “identical” runs of Design 1 and Design 2. Excel functions were used to estimate the mean and standard deviations of these values. Clearly, with only 10 runs of each design, our samples of the population were very small for statistical purposes and too much emphasis should not be placed on the exact values which emerged from our analyses.

Error Tolerance Analysis

Once the magnitude of thickness errors has been derived for a design, the effect of those errors on the performance of the design can be predicted with Error Tolerance Analysis. Again, we used the Essential Macleod for this purpose. The program allows two types of error to be defined: Mean Thickness Errors, which result in a consistent shift in performance, and Standard Deviation of Thickness Errors, which describes the random fluctuation of thicknesses about the mean.

In principal, mean errors can be eliminated by proper calibration of the process, but this is possible only after a large number of runs has been performed so that the true mean can emerge. In practice there will almost always be a small Mean Thickness Error.

Random Thickness Errors can only be reduced by improving control of the process. In the Tolerance Analysis, any number of “cases” can be generated, each case representing the possible performance of a coating run where thickness errors have been introduced by random generation within the mean and standard deviations defined. The number of “good” cases, as a percentage of the total number of cases, is an estimate of the process yield. Obviously the larger the number of cases, the more valid will be the predicted yield. We restricted ourselves to 20 cases to make the analysis reasonably manageable.

DESIGN 1 ANALYSIS

Layer thicknesses and indices for Design 1 are shown in Table II. The theoretical performance is shown in Figure 3 at both normal and 30^o angles of incidence. Note that the 0.15% bandwidth is ~ 1.04, which gives an early warning that thickness errors of the order of +/- 2% will cause problems with this design.

TABLE II: Design 1

LAYER	MATERIAL	INDEX	THICKNESS
	Air	1	Medium
1	SiO_2	1.46	878Δ
2	ZrO_2	~2.0	950Δ
	SF11	1.785	Substrate

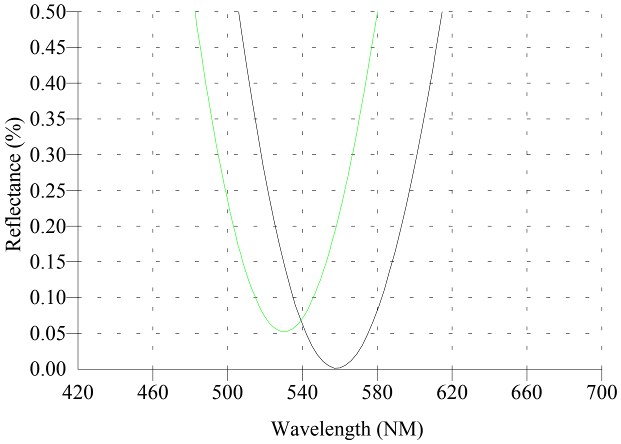

Figure 3: Theoretical Performance of Design 1 at 0^o & 30^o

Once it had become apparent that we did not have enough control with Design 1 to meet the requirements consistently, we stopped making adjustments and embarked on Phase I - 10 consecutive "identical" runs. The resulting reflectance spectra are shown in Figure 4. There are two groups: the set of plots at longer wavelengths is from 6 ^{o} measurements; the set at shorter wavelengths with somewhat higher reflectance is from 30^{o} measurements. There are obviously some mean thickness errors in that the reflectance minima are, on average, at wavelengths shorter than the target of 543nm. This is to be expected since we were only assessing repeatability at this stage. To get a rough idea of the process yield, we might ignore these mean errors and imagine that our target wavelength is, say, 530nm. Even then the yield from these ten runs would not be good: only 60% meet the 0.2% reflectance requirement and only 20% meet the 0.15% goal.

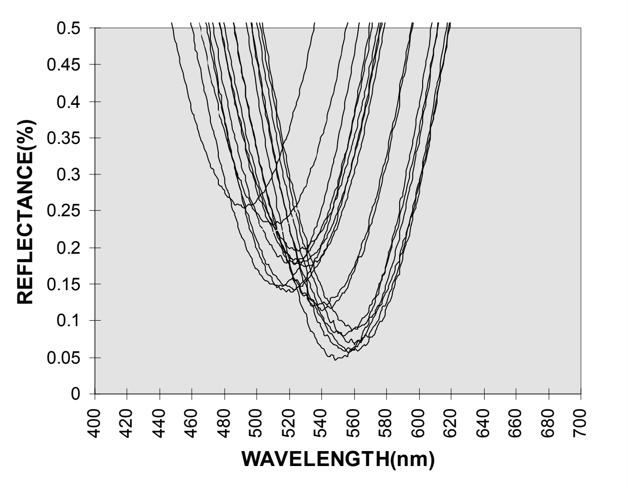

Figure 4: Measured Spectra from 10 runs of Design 1, at both 6E and 30E

The results of reverse engineering on these runs are shown in Table III. As expected, the thickness errors are quite large, with a standard deviation of 2 - 3% for both materials.

TABLE III: Reverse Engineering Results for Design 1

Material/Substrate	Mean Thickness	Standard Deviation
SiO_2 on Glass	869Δ	1.7%
SiO_2 on SF11	860Δ	2.5%
ZrO_2 on Glass	1009Δ	3.0%
ZrO_2 on SF11	986Δ	1.6%

Additionally, the ZrO_2 index exhibited an interesting behaviour which is shown in Figure 5. The index values for glass are consistently higher than for SF11, and a few data points are missing, but the trend is quite clear. Over the first 7 runs, the ZrO_2 index, as determined from glass results, drops rapidly from 2.06 before stabilizing at 1.97. For SF11, the corresponding values are from about 2.03 to 1.93. The SiO_2 index remained constant throughout the runs, with a value of 1.45.

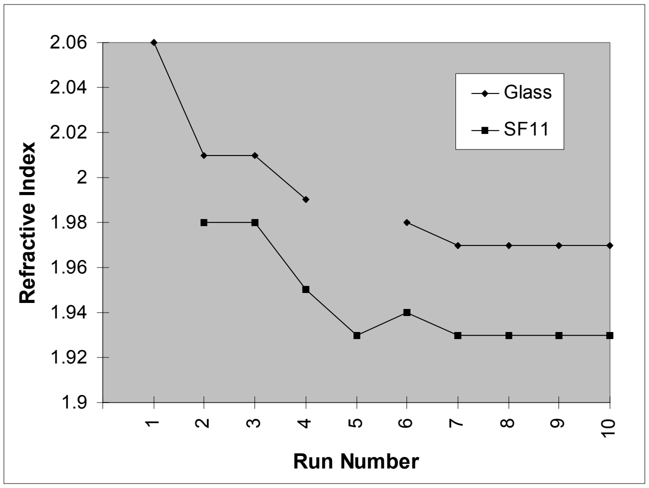

Figure 5: Evolution of Zirconia Index

One approach to improving the results with Design 1 would be to perform and discard the first 10 runs to allow the ZrO_2 to stabilize. However, this is not an attractive option from a manufacturing standpoint as valuable machine time is lost. Furthermore, even if the variation in ZrO_2 index is eliminated, two problems remain:

- The ZrO_2 index, after stabilizing, will be in the range 1.93 - 1.97, which is marginal for obtaining zero reflectance.
- The thickness errors by themselves are too large to allow the yield to reach 100%

Based on the reverse engineering results, an error tolerance analysis was done for Design 1 using the following values:

- 1% Mean Thickness Errors
- 2.5% Standard Deviation of Thickness Errors
- 0.5 error in the ZrO_2 index value

The resulting plots for 20 cases are shown in Figure 6. If we apply our customer requirement to these plots we get yields of 60% for 0.2%R and 25% for 0.15%R, values which are in good agreement with our original evaluation of the measured spectra in Figure 4.

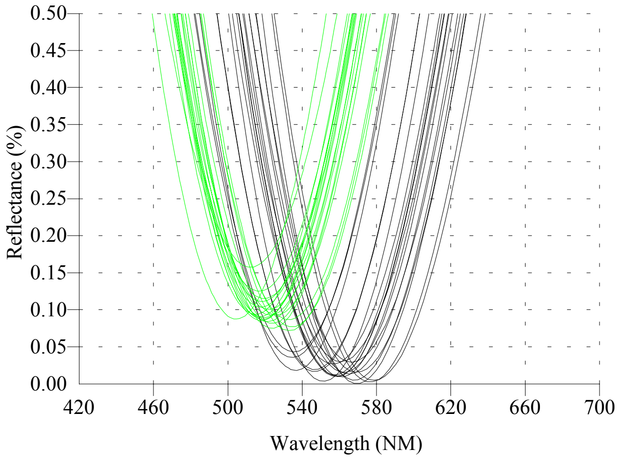

Figure 6: Error Tolerance Analysis for Design 1
Mean Error of 1%, SD of 2.5% and ZrO2 index of 1.95

In a sense, these results simply tell us what we already knew - that our level of control is not good enough to make Design 1 a viable process. However, the value of the exercise is that we have developed a theoretical model of the process errors which gives good agreement with measured results. We can now use the model to predict the yield which might be expected with other possible processes, such as Design 2.

DESIGN 2 ANALYSIS

Design 2 is summarized in Table IV and its theoretical performance is shown in Figure 7. The advantages of Design 2 were expected to be:

- Reduced sensitivity to thickness variations. Evidence of this can be seen in Figure 7, which shows that the 0.15% bandwidth is about 1.06, as compared to 1.04 for Design 1.

- Reduced sensitivity to index variations. From Figure 2, we can see that by increasing the high index (n_2) from about 2.0 to about 2.35, we have moved from a region where the thickness solutions are changing rapidly with n_2 to a region where the thicknesses are much less dependent on the value of n_2 .

- More consistent high index value

- Better source behaviour leading to smaller thickness variations. The Design 1 materials, SiO_2 and ZrO_2, are well known for their difficult behaviour. Neither flows well during evaporation and both tend to develop uneven surfaces which are not conducive to a consistent source characteristic. The Design 2 materials, MgF_2 and TiO_2, both form smooth puddles during deposition which should result in more consistent behaviour.

On the other hand, we were concerned about some possible problems with Design 2:

- The high index layer is very thin. It will be difficult to control and may exhibit inconsistent properties.

- The coatings may have poor durability because we can only heat the substrates to about 90EC.

These concerns turned out to be unfounded: although the TiO_2 layers did exhibit rather large thickness variations, the performance of Design 2 is quite insensitive to these variations. Durability was also surprisingly good - the Design 2 coatings all met the Adhesion, Moderate Abrasion and 24 hour Humidity requirements of MIL-M-13508.

TABLE IV: Design 2

LAYER	MATERIAL	INDEX	THICKNESS
	Air	1	Medium
1	MgF_2	1.38	1150Δ
2	TiO_2	2.35	92Δ
	SF11	1.785	Substrate

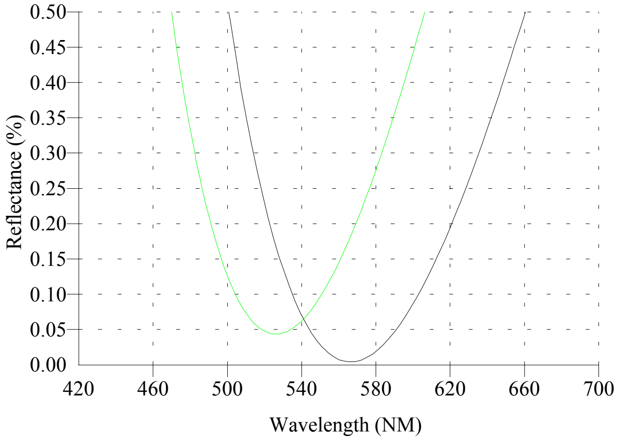

Figure 7: Theoretical Performance of Design 2 at 0E & 30E

The first step in Phase II was to run a tolerance analysis on Design 2 using the thickness errors established from Design 1. When we did this, the results were disappointing. Using thickness errors of 1% mean and 2.5% standard deviation, 20 cases of Design 2 gave yields of only 80% for 0.2%R and only 65% for 0.15%R. (Figure 8) These are much better than the yields for Design 1, but not nearly good enough to warrant setting up for prism coatings with Design 2.

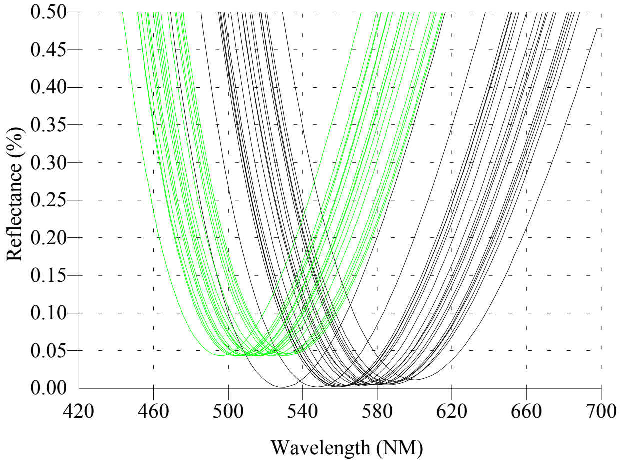

Figure 8: Preliminary Error Tolerance Analysis of Design 2: 1% Mean, 2.5% S.D., no index errors

At this point we might have been tempted to abandon the work in favour of a different approach, but luckily, before the results of the tolerance analysis were complete, we had already produced some sample coatings with Design 2. It was immediately apparent that the performance of Design 2 coatings was much more consistent than our analysis had predicted. After making a few adjustments to position the reflectance minima correctly, 10 consecutive runs were made with no changes and the results are shown in Figure 9. With one exception, all runs meet the 0.15% requirement at 543nm at both 6E and 30E. The "bad" run meets at 6E but reflects 0.17% at 30E.

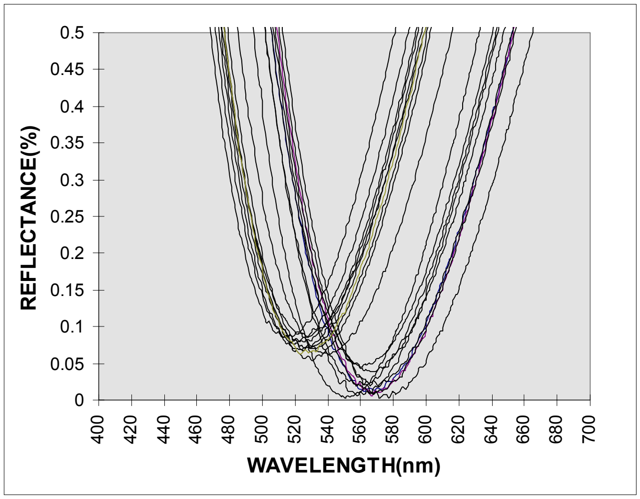

Figure 9: Measured Spectra of Design 2 at 0E & 30E

Obviously, in addition to reduced sensitivity, some other mechanism was at work in Design 2 which resulted in much better consistency. To find the explanation we reverse engineered the spectra from the 10 runs of Design 2 and the results are shown in Table V.

TABLE V: Reverse Engineering Results for Design 2

Material on Substrate	Mean Thickness	Standard Deviation
MgF_2 on Glass	1141Δ	0.6%
MgF_2 on SF11	1162Δ	1.0%
TiO_2 on Glass	132Δ	3.2%
TiO_2 on SF11	115Δ	4.2%

As expected, the variations in TiO_2 thickness are quite large - with a standard deviation of 3-4% they are even greater than for SiO_2 and ZrO_2. But the reason for the unexpectedly good consistency of Design 2 is now obvious: the MgF2 layer shows very small thickness errors, with a standard deviation of 0.6% on glass and 1.0% on SF11. These variations are almost a factor of three better than the equivalent values for SiO_2 in Design 1 (Table III). The reverse engineering revealed no significant index variations for either MgF_2 (n = 1.38) or TiO_2 (n = 2.35).

To confirm the viability of Design 2 we ran a tolerance analysis using the revised thickness error data (1% Mean and 1% S.D.) The resulting plots for 20 cases are shown in Figure 10, which looks quite similar to the measured data for Design 2 (Figure 9). Our version of The Essential Macleod does not allow the introduction of different sized errors for different layers in the tolerance analysis (a more recent option is available which does). Since the performance of Design 2 is dominated by the control of the MgF_2 layer, we used a 1% S.D. for both layers in the error modeling. This probably accounts for the slightly smaller spread of the predicted plots in Figure 10 as compared to the measured plots of Figure 9.

All 20 cases meet the 0.15%R requirement and so the predicted yield for Design 2 is now 100%. Table VI summarizes the predicted yields for the two designs with different error distributions.

TABLE VI: Predicted Yields from Tolerance Analyses

	Errors			Yield(%)	
	Mean(%)	S.D.(%)	Index	0.2%R	0.15%R
Design 1	1	2.5	0.05	60	25
Design 2	1	2.5	0	80	65
Design 2	1	1	0	100	100

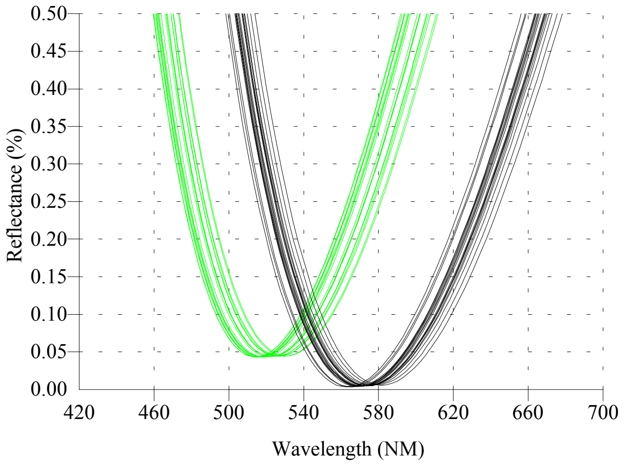

Figure 10: Error Tolerance Analysis for Design 2: Mean Thickness Error is 1%, S.D. is 1%, no index errors

Armed with confidence based on both real measurements and theoretical modeling, we set up and launched the manufacture of prism coatings. Since introducing the Design 2 process last year we have coated almost 80 prisms, each on two surfaces, with zero failures.

CONCLUSIONS

- A good coating design is one which takes manufacturing limitations into consideration, and which predicts high yields despite these limitations.
- Reverse Engineering and Error Tolerance Analysis can be used as valuable tools in our understanding of the coating process.
- In the particular case study described here, the stability of the MgF_2 layer emerged as the critical element in the success of the project.

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