Surface finish and microroughness of filled nylon injection mouldings

N J Elton

Introduction

This note illustrates the use of the Imaging Reflectometer for studies of filled nylon injection mouldings. For many plastic components, surface finish is an important factor in the acceptability of the finished product. Analysis by reflectometry can give useful insights into the structure of the surface layers to aid product development or production troubleshooting. The results reported here relate to a series of laboratory samples produced as part of a study on gate marking. As such they show a variety of surface markings due to gate marking (or ‘marbling’) and serve to illustrate the information generated by reflectometry analysis.

Instrument used

The instrument used was a standard SIRS75 Surfoptic Imaging Reflectometer with motorised x-y stage and mapping software. The standard parameters measured were refractive index, gloss, microroughness (roughness at a scale smaller than the wavelength of light) and macroroughness (roughness on a scale much larger than the wavelength of light, measured as the distribution of surface slopes).

Samples and measurements

Compounds of nylon 6 and a silane-treated calcined clay filler were made at four filler loadings, 10, 20, 30 and 40 wt%. An unfilled nylon was used as a benchmark. The compounds were injection moulded using a Arburg Allrounder 320M 750-210 (30 mm screw diameter) laboratory injection moulding machine. A standard test mould with one polished face was used which produced plaques approximately 80 x 80 x 2 mm. The compound formulation and process conditions were chosen as part of a study on gate marking and were designed to produce pronounced gate marking effects in the injection moulded plaques. Full details of the compounding and moulding conditions may be found in Legrix *et al* (2002).

Eight measurements were made at random points on the side of the sample corresponding to the polished face of the mould using the large measurement spot. Each measurement covers an elliptical area on the sample with major axes approximately 12 x 3 mm. Measurements were made in the injection direction.

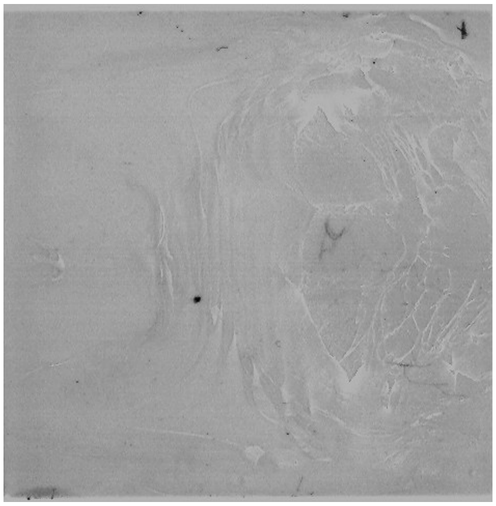

Figure 1a
Image of the 40 wt% filled plaque showing extreme gate marking effects. The gate is at the right centre of the image.

Figure 1b
Image of the 20 wt% filled plaque showing significant gate marking features. The gate is at the right centre of the image.

Average Properties

Figures 2a-c show the average results as a function of filler loading for the four main measurement parameters. Error bars on the plots represent the standard error of the 8 measurements.

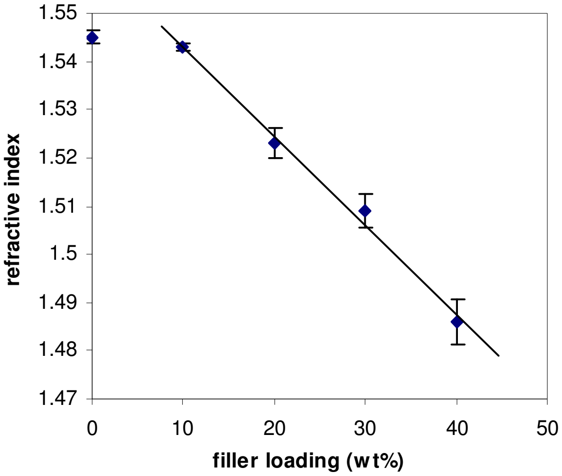

Figure 2a Refractive index vs filler loading.

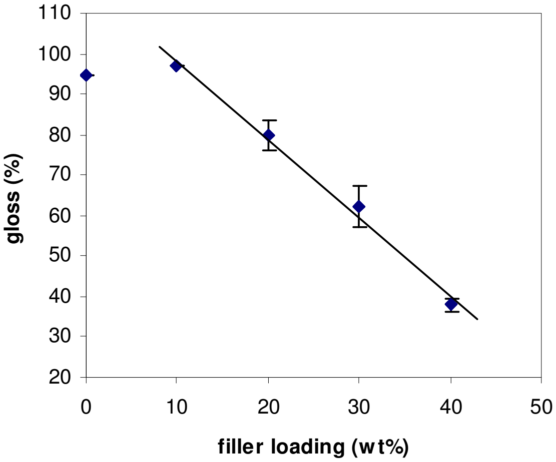

Figure 2b Gloss vs filler loading

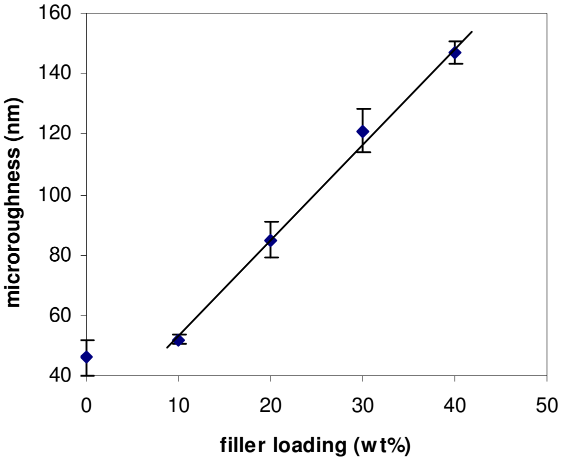

Figure 2c Microroughness vs. filler loading

All of the samples were macro-smooth. In each case the results for the unfilled nylon and that containing 10 wt% filler are essentially identical. Then, as the filler loading is increased from 10 wt% to 40 wt%, all the measured properties change almost linearly with increasing filler loading. The standard deviation of the results is greater for the samples with higher filler loading owing to greater point-to-point variation over the surface due to gate marking features.

The refractive index of the calcined clay filler is not known. That of hydrous kaolinite is about 1.55, very similar to the nylon (measured here at 1.545 ± 0.002). Assuming that the refractive index of calcined clay is similar to that of hydrous clay (for the solid mineral), then adding clay to the nylon compound would not be expected to change significantly the effective refractive index of the injection moulded plaque. The significant decrease in refractive index observed here is attributed to the increase in microroughness as discussed in the next section.

Relation of refractive index to microroughness

It is well known from ellipsometric studies that refractive index depends on surface microroughness. For materials with open surface porosity such as coated paper or paints formulated above the critical pigment volume concentration (CPVC), it is difficult to relate refractive index and roughness in an unequivocal way. However, the injection moulded plaques are formulated below the CPVC and open porosity is not expected. Therefore, it is practical to examine whether the observed changes in refractive index can be accounted for by the changes in microroughness. Ohlídal and Lukeš (1972a,b) provide a model, based upon scalar Kirchhoff theory, for the effect of random roughness on the complex refractive index. the effect of roughness on the ellipsometric parameters is governed by one parameter, $\tan\beta_0$, which is the rms surface slope, related to the rms roughness amplitude σ via the correlation length T, by $\tan\beta_0 = \sqrt{2}\sigma / T$. The model is valid for $\tan\beta_0 \leq 0.1$ and assumes that the rough interface lies between two homogeneous media (e.g. between a solid substrate and air). Using reflectometry it is possible to obtain the microroughness σ, but not the correlation length T. However, T can be used as an adjustable parameter. Figure 3 shows the observed change in refractive index with filler loading along with the prediction from the Ohlídal and Lukeš model for T = 3 µm. The excellent correspondence between model and observation suggests that the changes in effective refractive index for these samples are largely due to the changes in microroughness. Light reflected from the nylon surface “sees” the microrough surface as an effective medium being a mixture of air and nylon. As roughness increases, the surface layer where reflection occurs contains more air relative to solid nylon, leading to a reduction in the effective refractive index.

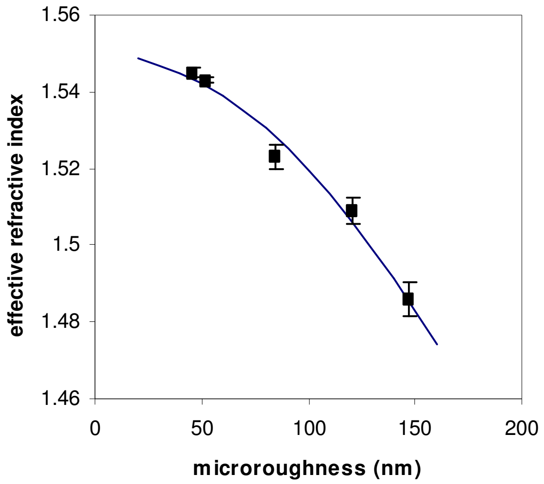

Figure 3
Change in refractive index with filler loading (squares) compared with prediction from Ohlidal-Lukes model (solid line). The good correspondence indicates that the observed decrease in refractive index may be attributed largely to the increase in microroughness (leading to increased entrainment of air at the surface).

Model parameters: RI of solid = 1.55, correlation length T = 3016nm.

Line Scans

Using the x-y stage, it is possible to obtain maps or line scans showing the spatial distribution of measured parameters over a surface. In the present example, it is interesting to examine how the surface properties vary with distance from the injection gate. Figures 4 a-c show line scans from the gate region to the back of the mould.

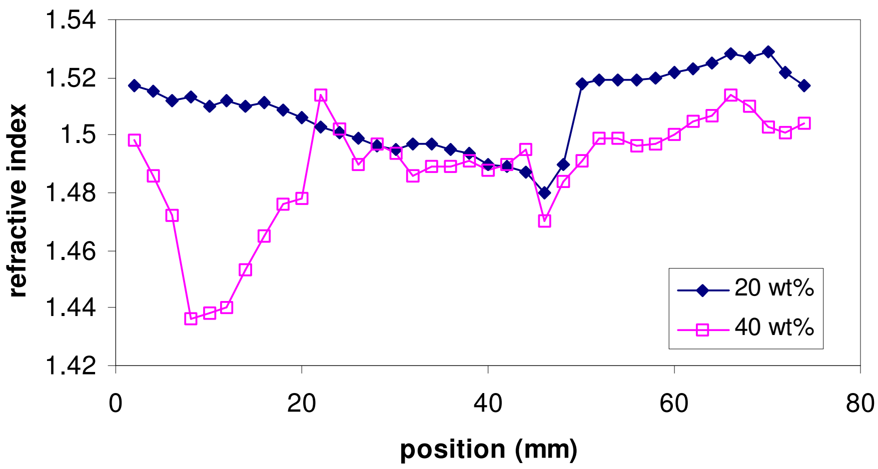

Figure 4a Refractive index. Gate end at position 75. The line scan was taken down the centre of the injection moulded plaque, averaging a strip approximately 6 mm wide. (The orientation of the line scan is the same as the photos in figure 1.)

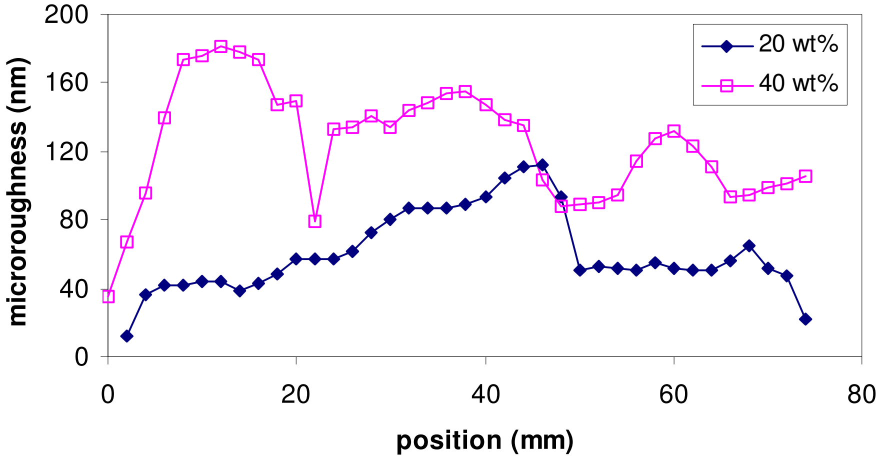

Figure 4b Microroughness

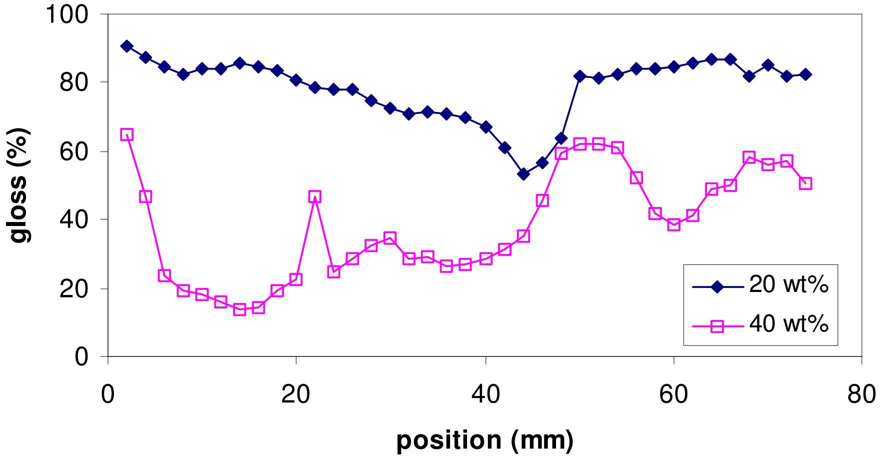

Figure 4c Gloss

The line scans corresponding to 0-10 wt% filled are relatively smooth (not shown). The data for 20 - 40 wt% filled specimens show more complex point to point variations reflecting the complicated gate marking patterns observed visually in the plaques. Note the very strong correlation between gloss and microroughness. In the more highly filled plaques, there is a tendency for the refractive index and gloss to decrease with distance from the gate. For these samples, microroughness increases with distance from the gate. Visual observation suggests that the region around the gate itself is “worse” in terms of gate marking features (c.f. Figure 1). However, reflectometery reveals that in fact it is the regions further from the gate that have lower gloss and increased surface microroughness roughness. Previous studies (Legrix et al. 2002) show the importance of viscosity and shear rate during injection and suggests that disruption and re-deposition of a partly solidified polymer skin may be responsible for the gate mark features. Some of the visually apparent markings may originate from sub-surface features.

Conclusions

Reflectometry can give insights into the surface optical and physical properties of plastics, including filled and injection moulded parts. The significance of such data may go beyond simply quantifying effects relating to visual appearance, because often mechanical properties like abrasion resistance, stain resistance and even impact strength can be directly related to surface structure.

Acknowledgements

Samples were kindly provided by Imerys Minerals Ltd, Performance Minerals Product Development Group, and thanks are due to Dr A Legrix for her comments on the present analysis.

References

Legrix A, Fugler A, Greenhill D, Goodman R and Paynter C 2002 Surface "marbling" in mineral filled nylon: origins and solutions. In: Proceedings of Injection Moulding 2002, Barcelona, 18th-19th March 2002, Paper 17, p.231-42. (Rapra Technology Ltd.)

Ohlídal, I and Lukeš, F 1972 Ellipsometric parameters of randomly rough surfaces *Opt. Comm.* **5**, 323-326.

Ohlídal, I and Lukeš, F 1972 Ellipsometric parameters of rough surfaces and of a system substrate – thin film with rough boundaries *Optica Acta* **19**, 817-843.