

Measurement Note

Assessment of Surface Characterisation Techniques for Adhesive Bonding

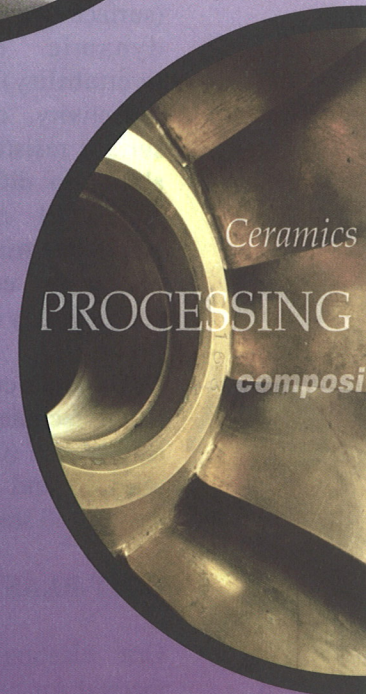
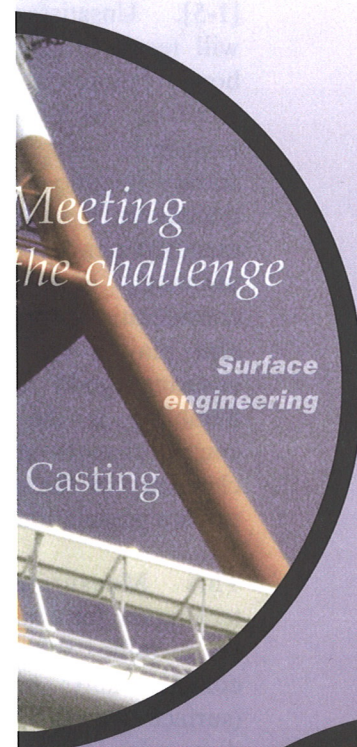
Surface preparation is recognised as the most critical step in the adhesive bonding process and considerable effort is frequently expended in optimising the surface treatment. Correct surface preparation is essential for good bond strength and maintaining the long-term structural integrity of bonded structures. Unsatisfactory surface preparation will result in premature and unpredictable interfacial failure of bonded joints. An essential part of ensuring good adhesion and long-term durability is the ability to monitor and control changes in surface morphology and chemistry of the substrate materials during surface preparation and storage.

This Measurement Note describes techniques for evaluating surface treatments of metallic substrates. These include: contact and non-contact profilometry (surface roughness), contact angle (wettability), micro-hardness, gloss/reflectivity, colorimetry, ellipsometry and surface resistivity. The techniques are assessed in terms of: ability to differentiate between treatments and level of treatment, suitability for laboratory trials or on-line inspection, ease of use, equipment costs, material compatibility and measurement uncertainty. Trials were conducted on grit-blasted, chromic acid etched and anodised aluminium alloys, and oil lubricated hot-dipped galvanized steel.

The research reported in this Measurement Note was undertaken as part of the Measurements for Materials Systems (MMS) Programme funded by the United Kingdom Department of Trade and Industry.

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AUGUST 2002



NPL 
National Physical Laboratory

INTRODUCTION

Good surface preparation is crucial to ensuring optimum bond strength and reliable long-term performance of bonded joints, particularly under hostile service conditions [1-5]. Unsatisfactory surface preparation will result in premature and unpredictable bond failure at the adhesive/adherend interface. As a result, considerable effort is frequently expended in optimising the surface treatment. The selection of surface treatments is usually based on adhesive manufacturers' recommendations, although technical, environmental and economic considerations (preparation costs and time), also play a role. An essential part of ensuring good adhesion and long-term durability is the ability to monitor and control changes in surface morphology and chemistry of the substrate materials during surface preparation and subsequent storage.

This Measurement Note describes techniques for evaluating surface treatments of metallic substrates. These include: contact and non-contact profilometry (surface roughness), sessile drop and dynamic contact angle methods (wettability), micro-hardness, gloss/reflectivity, colorimetry, ellipsometry and surface resistivity, are assessed in terms of ability to differentiate between treatments and level of treatment, suitability for laboratory trials or on-line inspection, ease of use, equipment costs, material compatibility and measurement uncertainty.

Trials were conducted using the previously listed techniques on grit-blasted, chromic acid etched (CAE) and anodised aluminium alloys, and oil lubricated hot-dipped galvanised steel.

GRIT-BLASTED ALUMINIUM

Grit blasting is generally the preferred method for removing rust, oxide layers, old coatings and heavy contamination from metallic surfaces. In addition to removing surface debris and roughening, grit blasting may cause physico-chemical changes, which affect surface energy and wettability [2-3].

The technique is fast and efficient, easily controllable and can be automated. Simple surface treatments, such as grit blasting are less prone to human error than other surface treatments (e.g. chemical etching) and therefore variation in bond strength tends not to exceed $\pm 20\%$.

A series of trials were carried out on 5251 aluminium alloy in which specimens were grit-blasted using 80/120 white alumina at a pressure of 85 psi for 0 (untreated), 10, 30, 60 and 120 seconds. The nozzle outlet was kept at a constant separation distance (150 mm) and normal to the sample surface. The objective of the exercise was to assess the ability of the various surface characterisation techniques to differentiate between the levels of treatment. A number of techniques were examined: (i) contact and laser profilometry and (ii) gloss/reflectivity.

Surface Profilometry

AG Electro-Optics carried out, on behalf of NPL, non-contact 3-D surface profile measurements on as-received (or untreated), and grit-blasted 5251 aluminium specimens. Specimens were also examined using a Surtronic hand-held contact surface roughness meter (diamond stylus with a radius of $5\text{ }\mu\text{m}$ - see Figure 2).

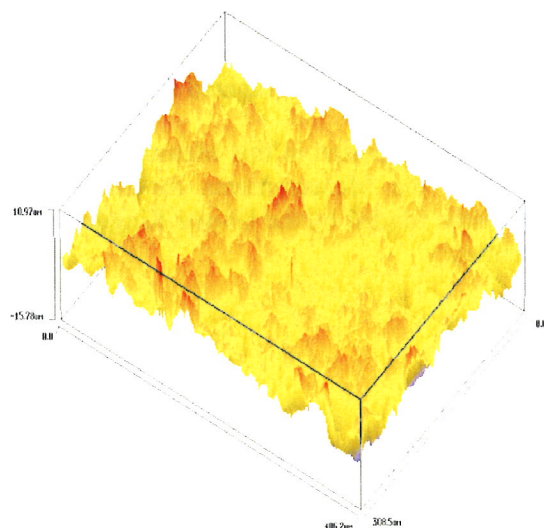


Figure 1: 3-D surface map of grit-blasted aluminium (courtesy of AG Electro-Optics).

Table 1: Surface Roughness Measurements on Grit-Blasted Aluminium

Treatment Time (secs)	R_a (μm)	R_q (μm)
Laser Profilometry		
0 (untreated)	0.20	0.25
10	2.05	2.67
30	2.36	3.03
60	2.60	3.30
120	2.37	3.01
Contact Profilometry		
0 (untreated)	0.24	-
10	1.83	-
30	1.79	-
60	1.98	-
120	2.08	-

The results presented in Table 1 show distinct differences in the values of R_a (average roughness deviation):

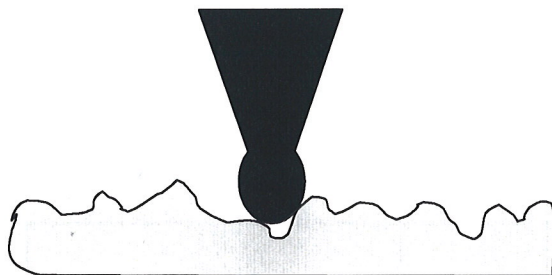
$$R_a = \frac{1}{L} \int_0^L |y(x)| dx$$

and R_q (root mean square (RMS) roughness deviation):

$$R_q = \sqrt{\frac{1}{L} \int_0^L |y^2(x)| dx}$$

for treated and grit-blasted surfaces with no indication of an intermediate state.

In the above relationships, L is the sampled length, x is the position along the sampled length and $y(x)$ are the roughness profile values.

**Figure 2: Contact (stylus) profilometry.**

The general trend was for surface roughness to remain almost constant with increasing treatment time. The results obtained for the two methods were in reasonable agreement, and hence either technique could be used to check for untreated regions.

Gloss/Reflectivity

A hand-held Rhopoint Surface Instruments Novo-Gloss gloss meter was used to measure surface reflectivity of the grit-blasted surfaces at two fixed angles (i.e. 20° and 60°). The instrument projects a beam of white light (filtered to give a spectrum response similar to that of the human eye) onto the target surface at a specific angle and measures the amount of specular reflected light. Surface roughness causes incident light to be scattered at other angles, such that the scatter increases with roughness. The instrument provides quantitative data on the uniformity and quality of the surface treatment [7]. The results (see Table 2) from tests conducted on the grit-blasted 5251 aluminium alloy show clear differences between treated and untreated surfaces, but no differences between the degree of treatment.

CAE ALUMINIUM

Chemical surface treatments (e.g. CAE) are intended to produce a strong, stable and chemically receptive oxide layer. This is achieved through a number of mechanisms including: (i) removal of weak boundary layer or transformation of boundary layer to provide a cohesively strong, stable oxide layer that is strongly adhered to the underlying substrate; (ii) increased surface energy (i.e. wettability) of the substrate surface; (iii) improved surface topography to enhance mechanical keying; and (iv) protection to improve environmental resistance of the substrate surface.

Table 2: Gloss Measurements on Grit-Blasted Aluminium

Treatment Time (secs)	Inspection Angle	
	20°	60°
0 (untreated)	121 ± 6	> 200
10	0.8 ± 0.1	2.9 ± 0.1
30	0.8 ± 0.1	3.1 ± 0.2
60	0.7 ± 0.1	3.0 ± 0.1
120	0.7 ± 0.1	2.9 ± 0.1

A series of trials were conducted on chromic acid etched 5251 aluminium alloy specimens. The specimens were immersed in CAE solution at 60°C for 0 (untreated), 10, 20, 30 and 60 minutes. On removal, specimens were sprayed with acetone, rinsed in cold water for 1 minute, rinsed in hot water for 2-3 minutes and finally air dried at 100°C in a circulating oven. The etching solution consisted of 37.5 grams of sodium dichromate dissolved in 500 ml of distilled water to which 75 ml of sulphuric acid was added. The solution was pre-heated for 30 minutes at 60°C.

The following techniques were used to examine the aluminium surfaces: (i) micro-hardness; (ii) contact and laser profilometry; (iii) atomic force microscopy; (iv) gloss/reflectivity; (v) colorimetry; and (vi) surface resistivity.

Micro-hardness

Micro-hardness testing involves smaller indenters and significantly smaller loads (10 mN to 10 N) compared with standard procedures (30 N or greater). The corresponding indentation is smaller and is associated with local properties of the surface rather than bulk hardness.

Standard hardness tests are unsuitable for assessing differences in surface layer properties. Micro-hardness tests can differentiate between soft and hard surfaces, however data variability increases with decreasing load. Indenter penetration needs to be less than 10% of the surface layer to avoid the influence of the substrate.

Vickers micro-hardness tests were carried out on CAE 5251 aluminium specimens. The test machine, which is fitted with a diamond tipped indenter, measures a comparative Vickers micro-hardness. The weight used for testing was 500 g. Differences in micro-hardness were minimal, ranging from 65.5 HV (Hardness Vickers) for the untreated material to 66.7 for 10 minutes exposure and 68.2 HV for 60 minutes exposure. The associated uncertainty was ±2 HV.

Surface Profilometry

Although differences in roughness exist between the untreated and CAE aluminium specimens, differences between the level of treatment are minimal (see Table 3). Again, the contact and non-contact profilometry methods produce similar results.

Table 3: Surface Roughness Measurements on CAE Aluminium

Treatment Time (mins)	R _a (µm)	R _q (µm)
<u>Laser Profilometry</u>		
0 (untreated)	0.20	0.25
10	0.26	0.35
20	0.31	0.42
30	0.26	0.35
60	0.28	0.40
<u>Contact Profilometry</u>		
0 (untreated)	0.24	-
10	0.19	-
20	0.31	-
30	0.30	-
60	0.38	-

Atomic Force Microscopy

Atomic force microscopy (AFM) proved successful in differentiating between levels of CAE treatment. The surface roughness parameter R_a was 73 nm and 266 nm for surfaces that had been treated for 10 minutes and 120 minutes, respectively. The corresponding R_q values were 1,258 nm and 2,682 nm. Surface profiles were measured using a Thermo-microscopes M5 AFM operated in contact mode. The AFM probe scans an area 100 mm square with loads typically of the order of 8 to 50 nN.

Gloss/Reflectivity

The gloss results presented in Table 4 show clear differences between CAE treated and untreated aluminium, and between levels of treatment. An increase in oxide thickness reduces surface reflectivity.

In order to optimise bond strength, surfaces are generally mechanically abraded (i.e. grit-blasted) prior to chemical treatment. Gloss measurements for grit-blasted + CAE and grit-blasted only material were not significantly different; as expected. R_a for grit-blasted surfaces is an order of magnitude greater than that for CAE and therefore dominates the response (see Tables 2 and 3).

Colorimetry

The spectral reflectance of CAE material was measured using a Datascolor Spectraflash 500 spectrophotometer. The spectrophotometer exposes a 10 mm diameter circular area on the surface to a light source with a daylight colour temperature and compares the percentage reflectance within the visible spectrum (360-750 nm wavelength) to that of reference white and black colour tiles.

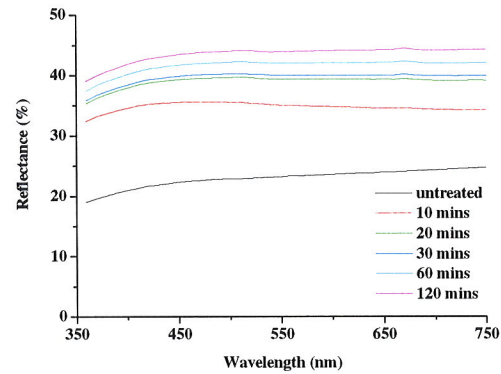


Figure 3: Reflectance spectra for untreated and CAE aluminium.

The colorimetry results shown in Figure 3 clearly indicate differences in surface reflectance between levels of CAE treatment. The reflectance increases with increasing oxide film thickness. As the oxide layer grows, the colour of the surface changes (i.e. becomes whiter). Colorimetry is able to differentiate between untreated and treated surfaces, and between short and long treatment times. It is difficult to differentiate between treatment times that are closely spaced. Also, consideration needs to be given to accurately measuring reflectance of the substrate prior to treatment. Variability in reflectance of surfaces may cause difficulties in data interpretation and quantification. Instrumented colorimetry systems are available for on-line inspection.

Surface Resistivity

Surface resistivity measurements were conducted on CAE 5251 aluminium alloy using an Industrial Development Bangor Ltd Model 482 surface resistance meter (see schematic in Figure 4). Flexible contour electrodes with a diameter of 75-80 mm were used to inspect 100 mm square specimens.

Table 4: Gloss Measurements on CAE Aluminium

Treatment Time (mins)	Inspection Angle	
	20°	60°
0 (untreated)	121 ± 6	> 200
10	35.7 ± 1.1	133 ± 3
20	30.6 ± 1.2	102 ± 1
30	15.6 ± 0.3	76 ± 1
60	9.3 ± 0.9	50 ± 2

The technique proved easy to use and could readily be adapted for on-line inspection, however it was insensitive to the levels of CAE treatment. The resistance for untreated aluminium was 1.5×10^7 ohms and $5-8 \times 10^5$ ohms for the different levels of exposure to CAE solution. Hence, the technique can be used to differentiate between untreated and CAE treated material, but not between levels of treatment. The resistance of the oxide layer, formed as a result of chemical treatment, tends to break down under prolonged exposure to an applied voltage.

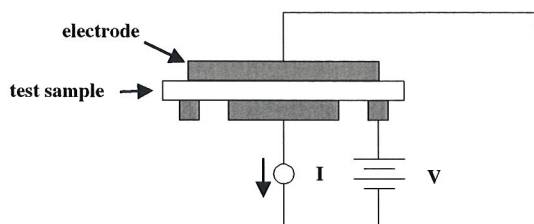


Figure 4: Surface resistivity equipment.

ANODISED ALUMINIUM

Alcan (Banbury Laboratories) supplied 6 sets of AA5754-O aluminium alloy samples (2.0 mm thick) with different surface treatments:

- 1) **Mill-finish** (a non-treated surface).
- 2) **Light clean** (lightly degreased to remove oils - most of the surface structure and oxides still remaining).
- 3) **Full clean** (an extensive electrolytic acid etch - equivalent of the optimised system without an anodised layer).
- 4) **Full clean + 25 nm barrier film** ("optimised" system with a full etch and a 25 nm barrier anodised film).
- 5) **Light clean + 25 nm barrier film** (a variant of (4) to give a lower level of etch).
- 6) **Full clean + over-anodised - braid** (a variant with a 25 nm barrier + 75 nm anodised film).

The surface properties of the six materials were measured using: (i) sessile drop and dynamic contact angle methods; (ii) contact profilometry; (iii) atomic force microscopy; (iv) gloss/reflectivity; (v) colorimetry; (vi) ellipsometry; and (vii) surface resistivity.

Dynamic Contact Angle (Wettability)

A Cahn Dynamic Contact Angle was used to measure the advancing θ_{adv} and receding θ_{rec} dynamic contact angles, and contact angle hysteresis ($\theta_{adv} - \theta_{rec}$) of samples less than 3g in weight. The analyzer employs the Wilhelmy plate technique (Figure 5) to measure forces exerted as the solid/liquid boundary is moved along the sample surface. Contact angle hysteresis is used to characterise surface heterogeneity and roughness. The sessile drop method was also used to measure contact angle. A FTA (First Ten Ångstroms) 200 Dynamic Contact Angle Analyzer was used for these measurements. The measurement process is automated, thus reducing measurement uncertainty.

Ideally, the contact angle should be reduced to a minimum in order to enhance adhesive strength. The lower the contact angle, the greater the tendency for the liquid to wet the solid, until complete wetting occurs at an angle $\theta = 0$. Large contact angles are associated with poor wettability.

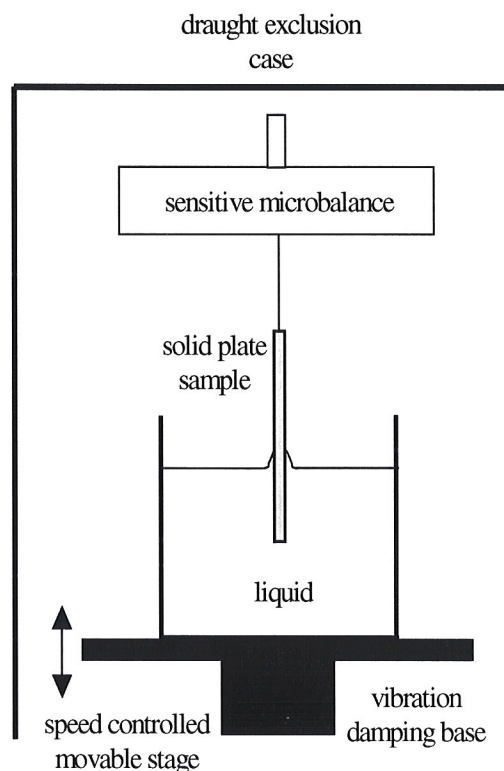


Figure 5: Wilhelmy plate method for measuring dynamic contact angle.

Table 5: Contact Angle Measurements for Anodised Aluminium

Dynamic Method	θ_{adv}	θ_{rec}	$\theta_{adv} - \theta_{rec}$
Mill finish	89.5 / 91.9	51.3 / 27.0	38.2 / 64.9
Light clean	84.5 / 84.8	0.0 / 0.0	84.5 / 84.8
Full clean	81.6 / 86.1	0.0 / 0.0	81.6 / 86.1
Full clean + 25 nm barrier	74.2 / 80.4	0.0 / 33.6	74.2 / 47.8
Light clean + 25 nm barrier	53.2 / 65.7	36.0 / 28.5	17.2 / 37.2
Full clean + 25 nm barrier + 75 nm porous	85.5 / 89.1	0.0 / 0.0	85.5 / 89.1
Sessile Method	θ_{static}		
Mill finish	96.0 $\pm$ 6.7 / 97.0 $\pm$ 4.1		
Light clean	98.3 $\pm$ 7.3 / 97.2 $\pm$ 4.2		
Full clean	53.7 $\pm$ 4.3 / 76.8 $\pm$ 3.5		
Full clean + 25 nm barrier	32.4 $\pm$ 6.2 / 42.1 $\pm$ 1.6		
Light clean + 25 nm barrier	83.3 $\pm$ 4.2 / 74.1 $\pm$ 1.7		
Full clean + 25 nm barrier + 75 nm porous	85.4 $\pm$ 2.8 / 86.7 $\pm$ 4.1		

Although the results obtained using the two methods were not identical, the results obtained using distilled water and glycerol probes tend to be self-consistent for each treatment. The sessile method measurements clearly indicate that the treatment identified as “optimised” (i.e. full clean + barrier layer) has the lowest contact angle, and hence improved wettability compared with the other surface treatments. Over-anodising the aluminium surface is counterproductive (i.e. contact angle similar to light clean and mill finish surfaces).

Care needs to be taken in preparing, storage and handling of specimens to prevent damage or contamination of the surfaces. Specimens need to be flat, with parallel faces and edges and free of surface or edge defects (e.g. scratches or burrs) and protected from contamination.

Surface Profilometry

The Surtronic profilometer was able to detect differences in roughness for the various surface treatments. Light cleaning produced higher surface roughness than the mill finish, whereas full cleaning reduced surface roughness. The addition of the 25 nm barrier layer further reduced the surface roughness. The addition of a 75 nm porous layer had no measurable effect on surface roughness. The operating range is 100 to 4000 nm, which implies that measurement reliability decreases as R_a approaches 100 nm (i.e. smooth surfaces).

Atomic Force Microscopy

AFM was less successful than profilometry in differentiating between surface treatments, although surface roughness was similar in

Table 6: Surface Roughness Measurements for Anodised Aluminium

Specimen	R_a (nm)	R_q (nm)
Contact Profilometry		
Mill finish	250	-
Light clean	390	-
Full clean	210	-
Full clean + 25 nm barrier	160	-
Light clean + 25 nm barrier	410	-
Full clean + 25 nm barrier + 75 nm porous	160	-
Atomic Force Microscopy		
Mill finish	89	133
Light clean	120	175
Full clean	124	177
Full clean + 25 nm barrier	161	249
Light clean + 25 nm barrier	214	288
Full clean + 25 nm barrier + 75 nm porous	166	237

magnitude to the corresponding contact measurements. There were no differences in roughness between light and full cleaned surfaces. The effect of the barrier layer was to increase surface roughness, but the 75 nm porous layer had no additional effect on surface roughness. Figure 6 shows a contour map of the “optimised” surface treatment.

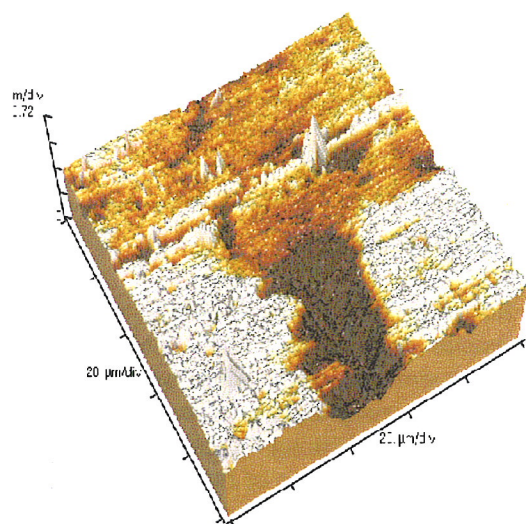


Figure 6: AFM surface map of “optimised” anodised aluminium surface.

Gloss/Reflectivity

Although there is a clear difference in reflectivity between the mill finish and the other surface treatments, it is difficult to differentiate between the various anodised treatments (see Table 7).

Colorimetry

Colorimetry proved successful in detecting differences in reflectivity between the various anodised treatments (see Figure 7). Again, there is a clear difference between the mill finish and the anodised surfaces. It is also possible to differentiate between light

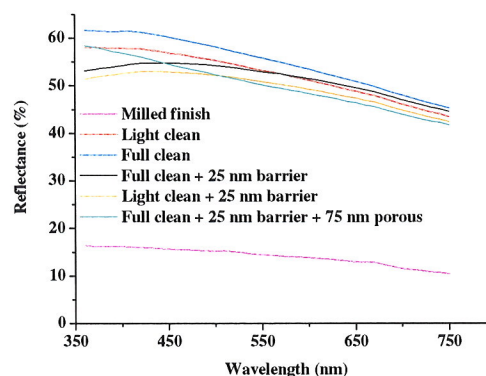


Figure 7: Reflectance spectra for anodised aluminium samples.

and clean surfaces, and between fully clean surfaces with and without a barrier layer. Cleaning increases reflectivity and the presence of the barrier layer reduces reflectivity.

The spectrum for the over-anodised surface was also different to the other surface treatments where reflectivity decreased linearly with increasing wavelength.

Ellipsometry

Ellipsometry uses polarized light to measure the thickness and refractive index of thin films [6-9]. The technique directs a polarised light beam onto a surface at an oblique angle of incidence and measures the phase change and intensity (amplitude) of the reflected light. Thin film thickness t , as well as optical constants n (index of refraction) and k (extinction coefficient, a measure of absorption) are derived from the measurement (complex index of refraction $\tilde{n} = n + ik$). Multiple reflections interfere to provide information on t , n and k for each surface layer interacting with the incident beam.

Table 7: Gloss Measurements for Anodised Aluminium

Specimen	Inspection Angle	
	20°	60°
Mill finish	91 ± 9	142 ± 8
Light clean	42 ± 3	115 ± 1
Full clean	46 ± 1	128 ± 1
Full clean + 25 nm barrier	40 ± 1	108 ± 2
Light clean + 25 nm barrier	48 ± 1	125 ± 1
Full clean + 25 nm barrier + 75 nm porous	37 ± 1	118 ± 1

The oxide layer thickness and optical properties (refractive index and absorbance) were measured using: (i) a single-wavelength ellipsometer; (ii) variable angle spectroscopic ellipsometer (VASE); and (iii) Infrared VASE or IR-VASE. VASE measurements were carried out by Alcan (Banbury Laboratories) on the anodised aluminium samples in order to determine the structure of the multi-layered oxide system present on each surface. Table 8 compares the results obtained using VASE with single-wavelength (courtesy of BP, Sunbury) and IR-VASE (courtesy of J A Woollam Co., Inc.). The VASE measurements were taken over the spectral range 193 to 2,200 nm, whereas the IR-VASE measurements were taken over the spectral range 2 to 30 μm . Both were recorded at multiple angles of incidence (70° - 75° by 5°). The beam diameter in each case was fixed at 6 mm. The fixed-angle single-wavelength instrument uses a He-Ne laser source with a wavelength of 632.8 nm and 45° polarisation.

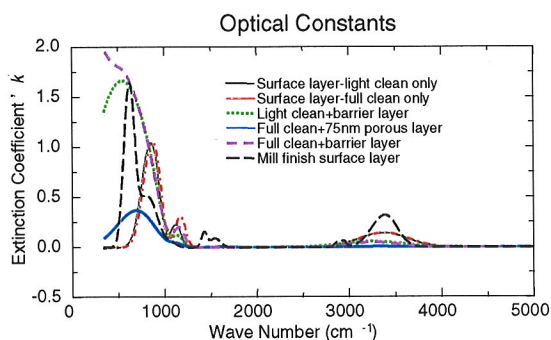


Figure 8: Molecular bond absorption IR-VASE data for anodised aluminium (courtesy of J A Woollam Co., Inc.).

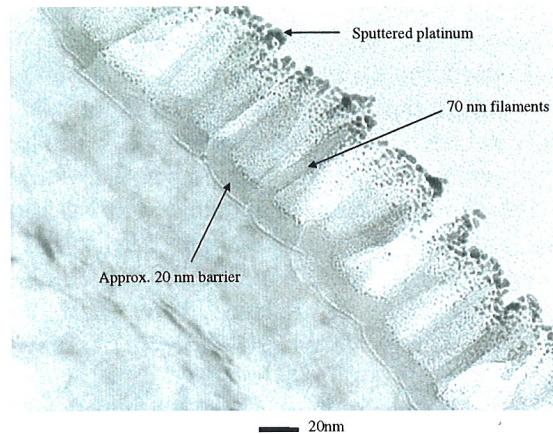


Figure 9: TEM image of the "over anodised" sample (courtesy of Alcan)

The molecular bond absorption bands for the different surface treatments were readily distinguishable in the IR ellipsometry data (Figure 8), therefore it was possible to finger print the surface chemistry (molecular bonds) present in the surface layers. Although the samples without a barrier layer have an oxide layer, it was impossible to determine the film thickness for these materials. Ellipsometry analyses requires a baseline substrate for the optical properties of the surface, which in the case of aluminium is an oxide layer. The technique will only measure properties of the additional oxide layer. Modelling the optical properties of multi-layered systems is a complex process, requiring a good knowledge of the optical properties of individual layers, and unless this information is available it is impossible to accurately determine the thickness of individual layers. Better knowledge of the optical properties of the oxide film may help explain differences in results.

Table 8: Ellipsometry Film Thickness Measurements for Anodised Aluminium

Specimen	Film Thickness (nm)	
	Barrier Layer	Porous Layer
VASE Measurements		
Full clean + 25 nm barrier	26	-
Light clean + 25 nm barrier	25	-
Full clean + 25 nm barrier + 75 nm porous	24	80
IR-VASE Measurements		
Full clean + 25 nm barrier	14	-
Light clean + 25 nm barrier	7	-
Full clean + 25 nm barrier + 75 nm porous	7	124
Single-Wavelength Fixed-Angle		
Full clean + 25 nm barrier	7-8	-
Light clean + 25 nm barrier	2-7	-
Full clean + 25 nm barrier + 75 nm porous	-	52

Transmission electron microscopy (TEM) was used by Alcan (Banbury Laboratories) to confirm the VASE results (see Figure 9). IR-VASE and single-wavelength ellipsometry results, presented in Table 8, were deduced from data obtained from all six materials with and without barrier and porous layers. It was assumed for modelling purposes that the surface layer had a refractive index close to Al_2O_3 ($n = 1.766$).

The presence of a barrier layer or contamination (i.e. light clean) results in an increase in molecular bond absorption (Figure 8). Mill finish material has the lowest absorption for any given wavelength. The single-wavelength technique was able to detect differences in oxide film thickness (Table 8) and the presence of contamination on the aluminium surface, although it was not possible to determine the refractive indices for the various oxide films. There was also evidence in all cases of differences in optical properties in the X- and Y-directions resulting from the milling process.

Surface Resistivity

Surface resistivity measurements were carried out on the aluminium samples by Industrial Development Bangor Ltd. The results were inconclusive, although it was possible to differentiate between a mill finish and the light and full clean surfaces without either a barrier or porous layer. The over-anodised surface recorded the highest resistance (1×10^8 ohms), whilst the light and full clean surfaces had the lowest resistance ($2\text{--}6 \times 10^5$ ohms).

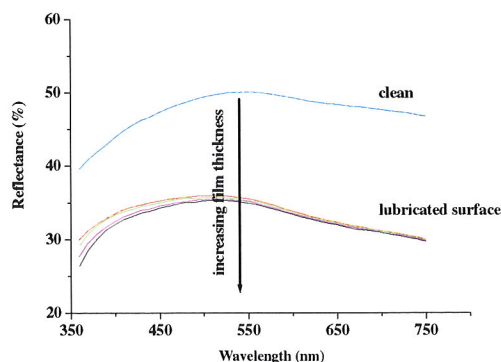


Figure 10: Reflectance spectra for increasing thickness of oil coating on galvanised steel.

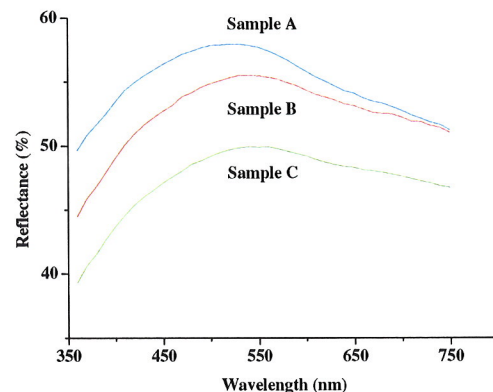


Figure 11: Variability in colorimetry reflectance for clean galvanised steel.

GALVANISED STEEL

A few new generation adhesives have been developed to bond to oily surfaces, thus eliminating the need for expensive surface treatments and enabling assembly in factory environments. In contrast to conventional adhesives, the presence of an oil lubricant does not significantly reduce adhesion when using these materials. A key issue relates to controlling the thickness and uniformity of lubricants on areas to be bonded.

Double-sided, hot-dipped galvanised steel (Zintec) with a gauge thickness of 1.5 mm (supplied by Corus) was coated in MP404 oil and examined using: (i) colorimetry; and (ii) ellipsometry. An electronic pipette was used to apply a known quantity of oil to the material, which was dispersed over the entire surface and allowed to settle for 24 hours to produce a uniform layer.

Tests were carried out on galvanised steel in which the amount of oil (i.e. thickness) on the surface was steadily increased. Ellipsometry proved ineffective in detecting differences in the amount of oil present on the specimen surface (as the film thickness was too large). Colorimetry was successful in detecting increments in oil film thickness (Figure 10). Shorter wavelengths seem to provide better discrimination. Colorimetry can be used to quantify the amount of oil present on an adherend surface, provided due care is taken when carrying out the measurements.

Table 11: Suitability of Characterisation Techniques for Different Surface Treatments and Substrates

Technique	Grit-Blasted	CAE	Anodised	Oil Lubricated	Corona Discharge ^[10]
Micro-hardness	No	No	No	No	No
Contact angle	Yes	Yes	Yes	No	Yes
<u>Profilometry</u>					
Contact	Yes	Yes	Yes	No	No
Laser	Yes	Yes	Yes	No	No
AFM	Yes	Yes	Yes	No	No
Gloss/reflectivity	Yes	Yes	possibly	No	No
Colorimetry	Yes	Yes	Yes	Yes	No
Ellipsometry	No	Yes	Yes	No	No
Surface Resistivity	No	No	No	No	No

The reflectance spectra obtained from clean galvanised steel prior to the addition of MP404 varied markedly from sample to sample (Figure 11), and hence indicate the need to inspect/map the entire surface to obtain background reflectance values prior to coating the surface. It is possible to subtract background reflectance data from the reflectance data obtained for the coated material.

It is difficult to quantify small reductions in reflectance due to increasing oil film thickness, and hence the total reflectance was summed over the entire spectral range of 350 to 750 nm. This is a convenient metric for quantifying the amount of oil on the adherend surface, thus enabling the user to directly relate the film thickness to a measurable parameter (i.e. reflectance). Figure 12 shows a comparison of total reflectance as a function of film thickness,

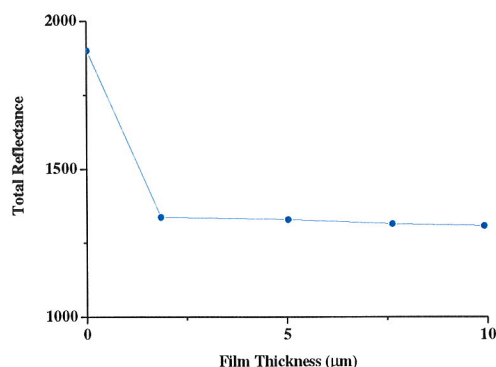


Figure 12: Total reflectance for clean and oil coated galvanised steel with increasing film thickness.

although the reduction is evidently small.

Very thin layers of oil can be expected to be difficult to detect and quantify, although ellipsometry is perhaps more promising for measuring the thickness of thin layers rather than thick layers.

CONCLUSIONS

Most of the techniques studied proved useful in determining changes in surface properties for different levels of treatment, although the range of application of these techniques to the systems studied was generally limited (see Table 11). The results clearly establish colorimetry as the most versatile technique for detecting changes in surface morphology. It was possible to quantify changes in reflectivity and relate these changes to levels of treatment or various stages in production of multi-layered systems. It is also adaptable for on-line inspection. Ellipsometry can be used to accurately determine layer thickness of multi-layered systems, provided the analyst has a good knowledge of the technique and optical properties of individual layers within the system. The sessile drop method proved far more reliable than the Wilhelmy plate technique in differentiating between surfaces with good and poor adhesion. The latter requires considerable care in specimen preparation, whereas there are no special preparation requirements for the sessile drop method. The portable units used for measuring surface roughness, surface resistivity and gloss required minimal operator training and were relatively inexpensive.

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Acknowledgements

The research reported in this Measurement Note was undertaken as part of the Measurements for Materials Systems (MMS) Programme funded by the United Kingdom Department of Trade and Industry. The authors would like to express their gratitude to AG Electro-Optics, Alcan International Limited (Banbury Laboratories), BP (Sunbury), Corus, Industrial Development Bangor Limited, J A Woollam Co., Inc., and to colleagues at the National Physical Laboratory, particularly Mr Bruce Duncan and Dr Giles Aldrich-Smith.

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