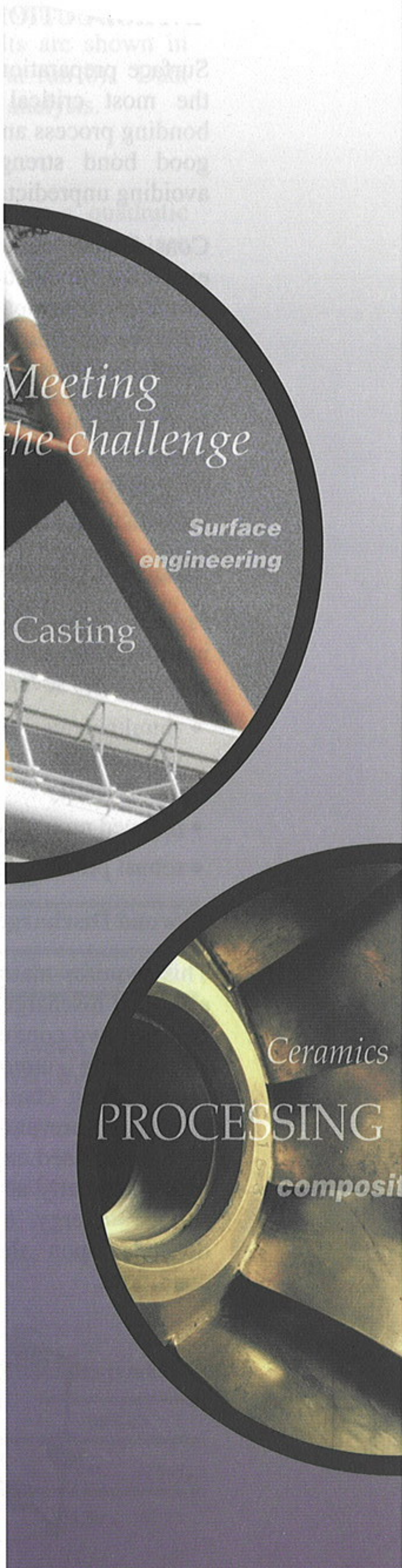
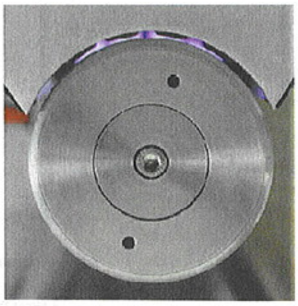
Measurement Note



Optimisation of Corona Discharge Surface Treatment for Improved Adhesive Bonding using Design of Experiments

Surface preparation is widely recognised as the most critical stage in the adhesive bonding process. Good surface preparation is essential for bond strength and durability. Conversely, poor surface preparation and treatment results in premature and unpredictable interfacial bond failure. Considerable cost and effort is frequently expended in industry, both to determine suitable treatment techniques and to optimise existing surface treatments through detailed process knowledge. This can be achieved using Design of Experiments to devise an economical matrix of trials. The trial conditions are uniformly distributed throughout the processing window of interest, for many variables simultaneously, and can be statistically analysed to give information on non-linearity, interactions and confidence limits for measured output responses/properties.

This Measurement Note presents the results from a simple study of the corona discharge treatment of glass-fibre reinforced polypropylene. The Design of Experiments approach was used to devise and analyse a small, statistically selected set of treatment trials; enabling the effect of the principal processing variables and their interactions to be investigated efficiently. The effects of the key parameters: input power, treatment time and electrode separation were considered. Dynamic contact angle analysis of the treated material was performed to evaluate the impact of these factors on the surface adhesion quality. A summary of the design strategy, implementation, observations, important results and principal conclusions of this investigation is provided.



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- treatment time mid-level value set to 0.2 not 0.22 as required
- electrode separations were grouped in a non-incremental order, not randomised
- trials in design were repeated not replicated.

IMPLEMENTATION

The material used was 4 mm thick injection-moulded discontinuous glass-fibre reinforced polypropylene plates with a glass fibre weight content of 50%.

The trials were carried out using a Sherman Treaters GX10 corona laboratory bench-top unit with a 280 mm wide U-channel metallic electrode.

Specimens for contact angle measurements were prepared, in order, as follows:

- 2 off 20 × 20 mm specimens per run were cut from two different mouldings giving two sets of nominally identical specimens
- cleaned thoroughly with isopropanol before treatment
- stored, and handled, on edge in a desiccator before and after treatment
- treated identically on both sides and within same region of corona treater (to avoid uncontrolled error due to varying field strength or changes in electrode separation with position).

Surface Contact Angle Measurements

A Cahn dynamic contact angle analyser was used to measure the contact angle on samples with a distilled water probe liquid, via the Wilhelmy plate technique [1, 2]. The lower the contact angle, the greater the wettability of the surface, which results in better adhesion. The results are shown in Figure 2 (untreated values at run 0). Data pairs were averaged prior to analysis.

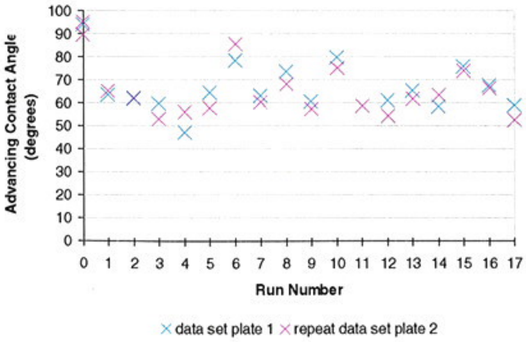

Figure 2 - Contact angle results.

ANALYSIS

The data were analysed using a quadratic model, the ANOVA results for which are given in Table 2 and the coefficient statistics in Table 3. Thus the power, treatment time and (treatment time)^2 showed a significant effect. The electrode separation did not, but this may have been due to the limited range studied.

Table 2 – Response Surface ANOVA Analysis

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-Value	H_0 %
model	1007.31	3	335.77	*57.26*	*0.01*
A	749.52	1	-	*127.82*	*0.01*
B	139.53	1	-	*23.80*	*0.03*
A^2	170.19	1	-	*29.02*	*0.01*
residual	76.23	13	5.86		
lack of fit	37.67	9	4.19	*0.43*	*86.27*
pure error	38.56	4	9.64		
total	1083.54	16			
Adj R-squared = 0.91				*Pred R-squared = 0.89*	

The process model relating contact angle, θ, to treatment time, A, and power, B, is:

$$\theta = 94.13 - 141.22\,A - 16.71\,B + 198.74\,A^2$$

This predicts an optimum surface treatment with a contact angle of 52.34°:

- treatment time = 0.36 min/m
- power = 1 kW.

Table 3 – Model Coefficient Statistics (coded)

	Coefficient Estimate	95%CI lower	95%CI upper
Intercept	60.15	58.38	61.93
A - treatment time	-9.68	-11.53	-7.83
B - power	-4.18	-6.03	-2.33
A^2	6.44	3.86	9.02

The closest trial to these settings was run number 4 which gave a contact angle of 51.48° for a treatment time of 0.4 min/m and a power of 1 kW.

Figure 3 shows the effect on the measured response of changing significant factors individually (left) or in combination (right) using the process model.

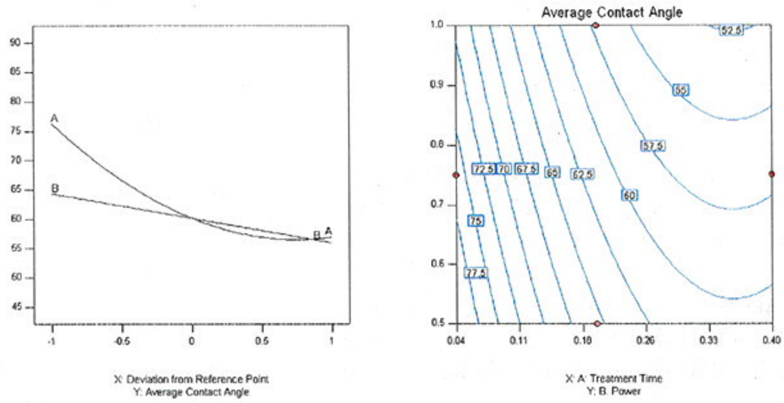

Figure 3 - Typical model output showing non-linear response; perturbation plot (left) and contour plot (right) for A: treatment time and B: power.

CONCLUDING REMARKS

The corona discharge trials demonstrated the methodology behind planning and conducting a test matrix in a systematic manner using the DoE approach. It was shown that an optimum process setting existed, giving a minimum contact angle and potentially improved adhesion for the material studied.

References

1. *W. R. Broughton and M. J. Lodeiro, "Review of Surface Characterisation Techniques for Adhesive Bonding", National Physical Laboratory Report MATC(A) 66, February 2002.*
2. *W. R. Broughton, M. J. Lodeiro, R. M. Shaw, E. Arranz and S. Gnaniah; "Assessment of Surface Characterisation Techniques for Adhesive Bonding", National Physical Laboratory Measurement Note MATC(MN) 24, August 2002.*

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