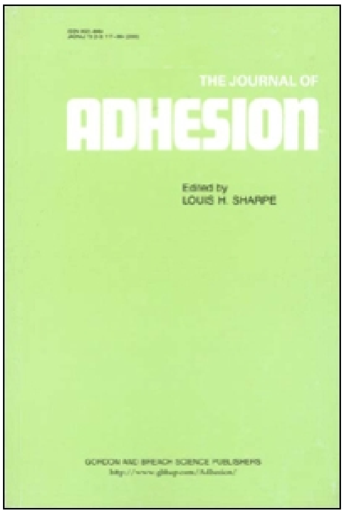

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Surface treatment of metal surfaces by corona discharge

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Aluminium and titanium surfaces have been treated by corona discharge in air and gave bonds of strength similar to those obtained by conventional chemical treatment.

INTRODUCTION

The use of high voltage electric discharges is well known for the treatment of some surfaces, mainly those of low surface energy, in preparation for adhesive bonding. Corona discharge at atmospheric pressures in air has been used for treatment of polyethylene surfaces for bonding or printing for nearly 30 years.[1,2] This has been extended to other poly-olefins as they have been developed for commercial use. An alternative treatment under reduced pressure in an inert gas has been proposed by Schonhorn (the "CASING" process).[3,4]

More recently, treatment with gas plasma has been suggested particularly for cleaning and removal of contamination from printed circuit components.[5] The technique has also been used in this Group for the treatment of printed cardboard surfaces and micro-electronic circuits on alumina substrates.

All the earlier work had been concerned solely with low energy, polymer surfaces but this last development involved treatment of a high energy, oxide surface. This led to a consideration of the general method for treatment of metals. Chemically, these are essentially oxide surfaces and there is little difference between the surface of aluminium metal and that of a pure alumina ceramic. Corona discharge in air at normal pressures had obvious attractions of simplicity for surface treatment and this has been explored.

MATERIALS AND METHODS

The metal adherends used were aluminium, clad alloy 2024-T3; and titanium, 6Al-4Va IMI 318A. The adhesive was Redux BSL 308A used with the corresponding primer Redux BSL-108 (Ciba Geigy Ltd.).

Conventional single lap joints were made between specimens 1 inch wide with a ½ inch overlap. In every case, immediately after the appropriate pre-treatment the surface to be bonded was painted with a thin layer of primer which was allowed to dry for 30 minutes at room temperature and was then baked at 120°C for a further 30 minutes. Joints were assembled with adhesive film in place and were cured in a press at 50 lb. in^{-2} and 170°C for an hour. The failing load of these joints were determined using an Amsler 10 ton, deadweight loading test machine. For each metal and for each treatment five replicate joints were made and tested. Mean failing loads recorded in these tests are given in Tables 1 and 2 together with their standard errors.

The cleanliness of the metal surfaces resulting from the various treatments was investigated by measuring the contact angles of drops of water on the surface using a reflecting goniometer. The mean values of these results are reported in Table 3.

SURFACE PREPARATION

Five different regimes were used in the treatment of each metal, four were identical for the aluminium and the titanium, but the ones involving a chemical treatment were different. The preparations used were:

As received — The metal was used without any treatment at all. Since it had been exposed to the atmosphere for some time and handled, it must have had at least a thin surface film of greases, hydrocarbons and possibly esters.

Solvent wiped — The area which was to be bonded was carefully wiped with a swab soaked in trichloroethylene and was allowed to dry for 20 minutes before further treatment or priming.

Corona discharge

The area to be bonded was treated for 10 minutes with a Corona discharge from a hand-held Edwards High Frequency Spark Tester Type ST4M providing up to 30 kV at 3.8 MHz.

Particular care was taken to ensure thorough treatment of the corners of the area where the stress would be concentrated.

Chemical treatment

For the aluminium, following solvent wiping, a chromic/sulphuric acid etch was used at 65°C for 10 minutes followed by thorough (20 minute) washing with distilled water and drying before priming.

For the titanium, following solvent wiping, a two stage treatment was used, first 30 seconds in a nitric acid (15% v/v), hydrofluoric acid (3% v/v) solution followed by 3 minutes in a trisodium phosphate (5% w/v), sodium fluoride (0.9% w/v) hydrofluoric acid (1.6% w/v) solution, all at room temperature. This was followed by thorough washing and drying in an oven at 66°C before priming.

RESULTS

TABLE I
Aluminium

Treatment	Joint strength kN. cm^{-2}	Type of failure % adhesive
As received	3.75 ± 0.08	68
Solvent wiped	4.16 ± 0.10	60
Chemical treatment	4.81 ± 0.04	42
As received + Corona discharge	4.73 ± 0.08	60
Solvent wiped + Corona discharge	4.99 ± 0.08	54

TABLE II
Titanium

Treatment	Joint strength kN. cm^{-2}	Type of failure % adhesive
As received	2.48 ± 0.35	69
Solvent wiped	4.89 ± 0.08	50
Chemical treatment	5.70 ± 0.08	22
As received + Corona discharge	5.23 ± 0.14	50
Solvent wiped + Corona discharge	5.70 ± 0.08	22

TABLE III
Surface cleanliness indicated by contact angles

Treatment	Contact angle with water Aluminium	Titanium
As received	80°	88°
Solvent wiped	23°	32°
Chemical treatment	0	0
As received + Corona discharge	0	0
Solvent wiped + Corona discharge	0	0

CONCLUSIONS

For both metals the first three sets of results, showing the effect of classical treatments, are exactly as would have been expected. Solvent wiping gave a cleaner surface which gave stronger bonds but both the cleanliness, as indicated by the contact angle with water, and the bond strength was significantly improved by the appropriate chemical treatment.

Corona discharge treatment on the dirty metal as received appeared to give a cleaner surface than had solvent wiping, and the bond strengths were greater than those achieved by solvent wiping although not always as great as those achieved after appropriate chemical treatment. If the corona discharge treatment was preceded by solvent wiping which might be presumed to remove the majority (but not all) of greasy contamination, then bond strengths were achieved very similar to those attainable with the appropriate chemical treatments.

These results suggest that a very important part of the usual chemical treatments depends upon their ability to provide a clean and entirely grease-free surface. Similar surface conditions may be achieved by use of corona discharge. This has obvious advantages in avoiding the disadvantages and risks associated with using and disposing of highly corrosive chemical solutions. Furthermore it might be applied to the surfaces of large structures where immersion in liquid would be difficult or impossible.

Whether this type of treatment has any effect on the nature of the oxide surface and how durable joints prepared in this way may be, remain for further investigation.

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