FRACTURE OF EPOXY BONDED DYNAMIC PEEL SPECIMENS CONTAINING INTERFACIAL LAYERS.

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Previous work has identified the presence of interfacial layers in silicate filled epoxy adhesive in combination with hot-dip galvanised substrates. The properties of the interfacial layers have been found to be significantly different to that of the bulk adhesive. EDX analysis of the layers showed that there is an absence of silicate fillers within the layers. Mechanical test results are compared for the samples made from both hot-dip galvanised and uncoated mild steel substrates in combination with heat cured epoxy adhesive. Dynamic tests were conducted using an falling-weight drop tower with forces measured using an instrumented tup and stationary wedge.

INTRODUCTION

The strength and durability of adhesive bonds is of great importance in many applications of modern structural adhesives. Failure of epoxy resins is often due to crack initiation at or near the interface and the substrate. Pao (1), Knollman (2) and Compton (3) have identified interfacial layers between aluminium substrates and epoxy adhesives which differ in chemical composition and mechanical properties from the bulk adhesive. Previous work by the authors (4) has identified filler barren layers between a calcium silicate filled adhesive and both galvannealed and hot dip galvanised steel substrates. Pao (1) has shown that in single lap shear bonds, using aluminium substrates, the stress intensity factors and fracture parameters of interfacial cracks are strongly influenced by the ratio of the Young's modulus of the interfacial layer and that of the bulk adhesive.

The bonding of automotive structures constructed from galvanised steel sheet is currently being evaluated. The possibility of similar interfaces forming between such substrates and epoxy adhesives obviously exits. This work aims to

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determine whether similar interfaces exist between hot dipped galvanised steel substrates and a commercially available high-strength, single-part, hot-curing epoxy adhesive and to assess the effect of any such interfaces on the mechanical properties of single lap shear joints and the impact strength of such metal to metal bonds.

METHOD

Multiple bonds were prepared using commercial grade hot dipped zinc coated steel sheet. The adhesive used was a high strength single-part, heat-curing, calcium silicate filled epoxy. Lap shear bonds were assembled and cured according to the adhesive manufactures' instructions. In order to determine whether interfacial regions existed between the substrate and the adhesive, sections were cut across bonds. After sectioning the specimens were mounted in a cold curing resin prior to being ground and polished. The polished specimens were examined using optical and scanning electron microscopy and energy dispersive X-ray analysis. Once areas of interfacial layer had been identified, the Vickers hardness number for layers and bulk adhesive were determined using a Reichert microhardness tester.

In order to assess the effect of the interfacial layers on the single lap shear strength of metal to metal bonds specimens were produced from both the galvanised hot dip and mild steel sheet on which layers do not form. In order to ensure that the results from the two sets of samples would be directly comparable, the mild steel samples were prepared by removing the Zn-Fe surface layer from the hot dipped galvanised sheet using 50% HCl. All substrates were prepared by lightly abrading with fine emery paper and degreasing with alchohol prior to assembly and cure. The cured bonds were then tested to destruction at a cross head displacement rate of 5mm per minute in a tensile testing machine. The resulting fracture surfaces were examined using stereo optical microscopy.

The impact performance of adhesively bonded joints and structures is obviously of great importance to automotive applications and for this reason impact peel test results for samples constructed from both the galvanised and un-coated mild steel (prepared as above) were obtained. The tests were conducted using an instrumented falling weight drop tower, forces being measured using an instrumented tup, travelling with a velocity of 2 ms-1 and stationary wedge, in accordance with ISO 11343. Again the resulting fracture surfaces were examined using stereo optical microscopy.

RESULTS AND DISCUSSION

Figure 1 shows a secondary electron SEM image of a section through a galvanised steel bond, the interfacial layer [2] is clearly visible between the zinc coating [3] and the bulk adhesive [1]. Energy dispersive X-ray analysis showed the interfacial layer to be barren of the calcium silicate filler found within the bulk adhesive. The dot mapping for Si and Ca is overlayed in figure 1. The micro hardness of the bulk adhesive was found to be 1.85 Hv ± 0.11 compared with 4.59 Hv ± 0.45 for the interfacial layer, showing that the interfacial layer is significantly harder than the bulk.

Static Tests. Force/displacement characteristics for lap shear tests show that fracture occurs at a lower level of force and extension in the galvanised sample than in the case of the mild steel substrate. It is also evident that significant plasticity occurred of the mild steel substrate, resulting in a much larger energy to fracture (31J) than in the case of the galvanised substrate which appears to fail in a brittle manner (9J). Observation of the resulting fracture surfaces, figures 2 and 3, shows that both failures are predominately cohesive and that the nature of the failure in the case of the galvanised substrate is considerable more brittle than the that in the case of the mild steel substrate. The fracture of the galvanised sample is continuous along one interface, while that of the mild steel initiates on one interface an alternates to the opposite interface. The results appear to be consistent with the formation of a hard brittle interface forming between the zinc layer and the bulk adhesive.

Dynamic Tests. Initial impact peel results show more clearly show a sensitivity to test speed and enhances the difference between the mild steel and galvanised substrate. The bond substrates of the mild steel impact peel specimens suffered from considerable bending while the galvanised specimens showed no sign of deformation.

In figure 4 the fracture load for the mild steel specimen rises sharply and then stays approximately constant for some time followed by a sequence of less sharp rises in load until the specimen fractures completely. The load peaks occur due to initiation of a fracture which then arrests after propagating rapidly at a lower load in each case. The fracture surface exhibits stress whitening at each point of crack initiation corresponding with an increase in load. The total fracture energy of this specimen was found to be approximately 10 Joules

Figure 5 shows a sample load trace for a galvanised specimen which shows a low fracture load with increasing propagation. In this case there is little increase in load during propagation and the crack appears to propagate rapidly almost constant load. The total fracture energy in this case was found to be approximately 3 Joules.

The fracture surfaces from samples of the impact peel tests in figures 6 and 7 show that fracture was always cohesive in the mild steel specimens and interfacial in the galvanised steel. Some small regions of failure in the galvanised layers were also seen in the galvanised samples, these show as bright white patches on the fracture surface. The fracture surface from the galvanised substrates are also dominated by bubbles in the interfacial layer. Similar bubbles are absent in the specimens with a mild steel substrate, suggesting a reaction occurring between the curing adhesive and the zinc coating of the galvanised steel.

Bubble formation was suppressed in one sample by curing the bond under a pressure of 2 bar. The energy absorbed in this case was also reduced but only to 6 Joules, which although higher than that without pressure, is still much lower than the mild steel substrate.

CONCLUSIONS

- Filler barren regions have been found to exist at the interface between hot-dipped galvanised steel substrates and a high performance, calcium silicate filled epoxy adhesive.
- These layers were found to be significantly harder than the bulk adhesive.
- Single lap shear tests show a significant reduction in energy to fracture in the galvanised steel samples.
- Fracture surfaces from both static and dynamic tests indicate a more brittle failure in the case of the galvanised steel samples.
- The formation of bubbles at the interface of the galvanised substrates does not explain the reduction in energy seen in the tests.

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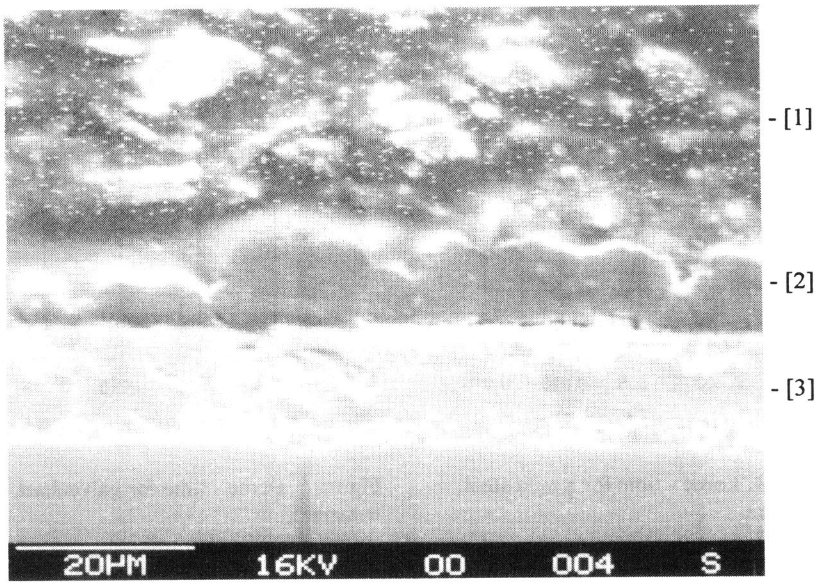

Figure 1. SEM image of the interfacial layer.

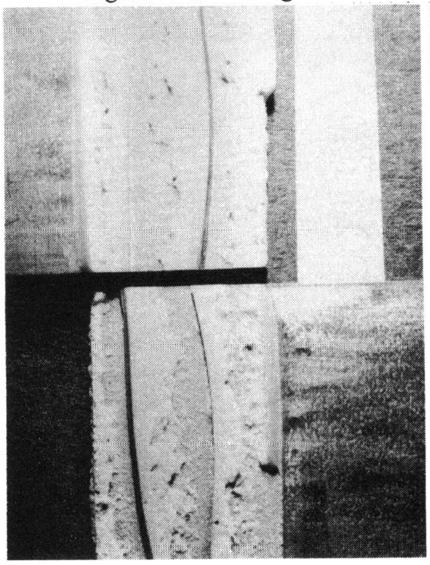

Figure 2. Fracture surface from static test on mild steel.

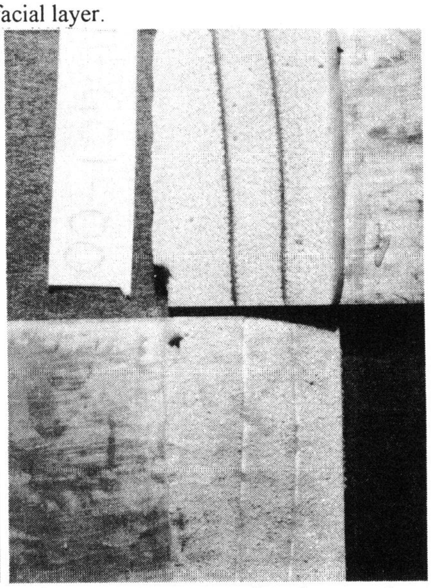

Figure 3. Fracture surface from static test on galvanised steel.

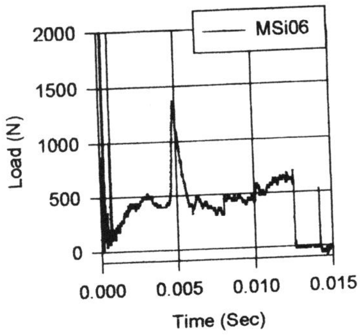

Figure 4. Force - time for a mild steel substrate.

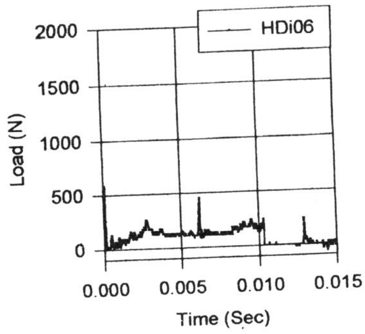

Figure 5. Force - time for galvanised substrate.

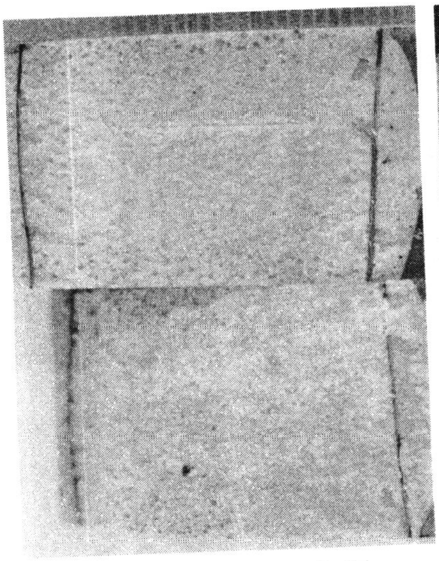

Figure 6. Cohesive fracture of mild steel substrate.

Figure 7. Interfacial fracture of galvanised substrate.