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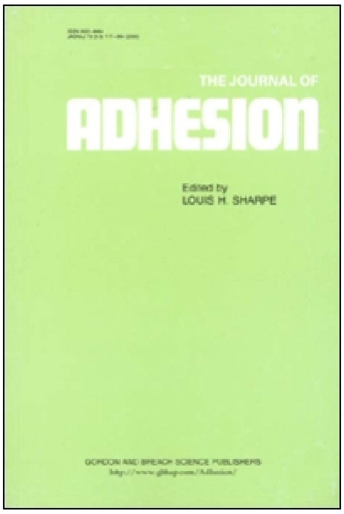

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# Influence of the Bonding Solvent on the Structure and Strength of Solvent Welded Joints

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# Influence of the Bonding Solvent on the Structure and Strength of Solvent Welded Joints

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The influence of the bonding solvent on the strength of solvent welded joints has been studied. Strong solvent welds are produced with solvents having the greatest ability to dissolve the polymer and not with solvents which could diffuse most rapidly into the adherend. The formation of a gel layer of highly mobile chains (on the application of a good solvent to the mating surfaces) promotes extensive and intimate bonding across the original interface so that no plane of weakness is obtained. Such welds exhibit high strength since they do not have any preferred plane of failure and extensive deformation of the weld accompanies crack initiation and propagation. When a poorer bonding solvent is used, solvent welds with lower strengths are obtained since soft and weak interfacial layers form at the original interfaces of the weld.

KEY WORDS Crack propagation; fractography; interphase structure; joint strength; solvent bonding; solvent welding.

## INTRODUCTION

There have been few studies[1–3] on solvent welding despite the fact that solvent welding is an important jointing technique for plastics especially in plastic pipeline systems. In particular, little is known about the influence of bonding solvents on the strength of solvent welded joints. Due to this lack of knowledge, commercially available solvent cements seem to be formulated empirically based on experience.

Titow and co-workers[1] were concerned with the extent to which bonding solvents may be removed from solvent welded polycarbonate joints after different drying schedules. Titow *et al.*,[1] found that residual solvent always exists and was concentrated within a small zone at the original interface. Studies[1] on the amount and distribution of residual solvent were carried out because these factors may affect the physical and mechanical properties of the weld and hence affect joint strength. Cherry and Yue[2] identified the physical properties of the adherend and 'adhesive', and the geometrical parameters of the solvent weld which together affected the joint strength. However, the strength of solvent welded joints would also be affected by the structure of the joint which, in turn, is controlled by the nature of the bonding solvent-polymer interactions and the nature of the polymer chain interaction across the mating surfaces. Both of the above factors would be dependent on the bonding solvent used.

Thus, the purpose of the present work is to investigate how some bonding solvents interact with the polymeric adherend to give welds of different structure, to investigate how the crack paths depend on the structure of the weld, and to investigate how the weld structure and the crack path affect joint strength.

Menges *et al.*,[3] and Cherry and Yue[2] who used bonding solvents which contained more than 15 wt% dissolved polymer all found that, in addition to the solvent affected zones (SAZ) observed by Titow *et al.*,[1] a soft residual final adhesive layer (FAL) existed in their solvent welds. The SAZ consist of portions of the adherend adjacent to the original interfaces that have been swollen and plasticized by the bonding solvents. The FAL was sandwiched between two SAZ. Titow *et al.*,[1] reported that there was no particular preferred plane of failure in their solvent welds which were tested in shear. However, Cherry and Yue[2] reported that failure always occurred within the FAL and along the FAL/SAZ interface.

The free volume of an amorphous polymer may be regarded[4] as a great number of channels and holes of molecular dimensions. Hence, when the polymer is in contact with a solvent, diffusion can occur almost immediately since the solvent molecules may occupy the empty holes and channels. A soft swollen layer forms[5,6] at the polymer surface as a result of this diffusion. When enough solvent has been absorbed by the swollen layer, a gel forms. The swollen and gel layers only form when solvents used have solubility parameters close to that of the polymer (*i.e.*, good solvents of the polymer). Such solvents have the same effect as plasticizers.[7] Hence the swollen and gel layers contain mobile polymer chains, the chain mobility being higher in the latter. Different bonding solvents produce swollen and gel layers with varying chain mobility due to their different solvent-polymer interactions.

The gel layer is the tacky substance that forms on the surface of amorphous polymers that have been attacked by good solvents of the polymer. If more liquid is absorbed, the gel layer will be diluted. Dissolution of the polymer can occur[4] through the dispersion of the diluted gel layer in the solvent.

## EXPERIMENTAL

### Materials

Four bonding solvents were used, namely, tetrahydrofuran (THF), methyl ethyl ketone (MEK), dichloromethane (DCM) and cyclohexanone (CYH). All the solvents were of analytical grade, and their specifications are given in Table I. The solubility parameters and the hydrogen bonding parameters in Table I were obtained from the data of Vincent and Raha.[8] The solvents were selected because

TABLE I
Specifications of bonding solvents

| Solvent | Tetrahydrofuran (THF) | Methyl-ethyl ketone (MEK) | Dichloro -methane (DCM) | Cyclohexanone (CYH) |
|---|---|---|---|---|
| Molecular weight | 72.11 | 72.11 | 84.93 | 98.15 |
| Boiling point | 67 | 79.6 | 40 | 155.15 |
| Density (g/cc) at 20°C | 0.8865 | 0.8045 | 1.316 | 0.9465 |
| Molar volume (cc/mole) | 81.34 | 89.63 | 64.54 | 103.70 |
| Solubility parameter $(\mathrm{cal/cm^3})^{1/2}$ | 9.5 | 9.3 | 9.9 | 9.7 |
| Hydrogen bonding parameter $(\mathrm{cal/cm^3})^{1/2}$ | 12.0 | 10.5 | 1.5 | 13.7 |

their solubility parameters are close to that [9.53 (cal/cm$^3$)$^{1/2}$] of polyvinyl chloride[7] (PVC).

Adherends were cut from 1.5 mm thick transparent PVC sheets supplied by a local company, Tin's Industrial Co. Ltd. The modulus of the PVC sheet was determined as 3.11 ± 0.06 GPa from tensile tests in an Instron machine using an extensometer.

### Solvent diffusion experiments

Preliminary dissolution tests involved the immersion (for 24 hours) of two grams of PVC sheet in sealed beakers containing 50 ml of the individual bonding solvents. Complete dissolution of the PVC was observed in the beakers containing THF and CYH. The few particles of PVC in the beaker containing MEK completely dissolved on light stirring. All the PVC in the beaker containing DCM was swollen but not tacky and did not dissolve even with vigorous stirring.

The rate of weight gain was determined by suspending preweighed specimens in the solvent bath under room temperature (24°C) conditions. After immersion for selected times, and after the excess solvent had been quickly wiped off with a tissue, the specimens were weighed on an electronic balance. To minimize errors, a different specimen was used for each point on the weight gain graph. Specimens with dimensions of 40 × 20 × 1 mm were used in order to maximize the major surface with respect to the thickness so that errors arising from diffusion at the edges could be neglected.

### Solvent evaporation experiments

The rate of free evaporation of each bonding solvent (under room temperature conditions) was determined by measuring the rate of solvent loss from a 100 ml beaker with an exposed solvent surface area of 18.5 cm$^2$. Each beaker contained 100 ml of solvent and was placed on an accurate (± 0.0001 g) balance so that the solvent evaporation in a still air environment could be monitored constantly. However, the rate of solvent evaporation from the SAZ for each solvent was determined with specimens which had been immersed in the given solvent for one hour. The dimensions of the specimens used in the latter experiment are similar to those used in the weight gain experiments.

### Tensile and microhardness tests

A double-lap joint configuration was used to minimize the effect of adherend bending. Each adherend was washed with detergent, rinsed, and wiped dry before being solvent welded. During solvent welding, the amount of solvent applied to the mating surfaces was kept constant. Unless specified otherwise, all specimens had overlap lengths of 35 mm and were dried for 5 hours before they were tested in tension in the Instron testing machine. Details of the specimen preparation for microhardness testing are outlined elsewhere.[2] The microhardness measurements were determined in a Leitz microhardness tester using a load of 10p (0.0981 N) and a loading time of 15 sec. The reciprocal of the size of the indentations was taken to be the hardness.

### Weld structure and fractography

The structure of the as-prepared solvent welds obtained using the different bonding solvents was determined utilizing a technique which is described in detail elsewhere.[2]

The locus of failure in solvent welded joints could be determined by examining specimens with overlaps of greater than 35 mm (which had been dried for 24 hours) in which the central adherends necked during testing. Testing of such specimens was stopped after the necks, which initiated at the edge of the overlap, had propagated no more than 10 mm into the solvent welded joint. The necked specimens were then polished metallographically to 3 μm and examined under a microscope to study the crack paths in the solvent welds.

The fracture surfaces of the sheared solvent welded joints were sputter coated with gold and examined in the scanning electron microscope.

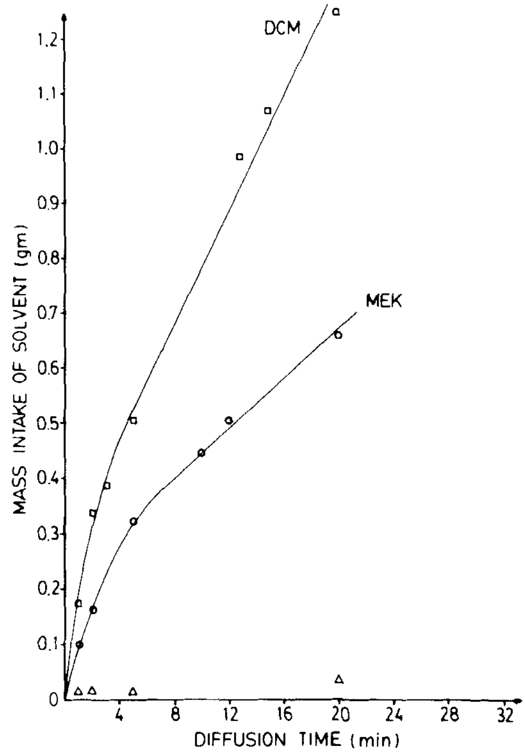


FIGURE 1 Rate of solvent intake by adherend.

## RESULTS AND DISCUSSION

### Solvent diffusion and evaporation

The rates of solvent intake by the PVC are shown in Figure 1 for DCM and MEK. From Figure 1, it can be seen that for the initial period of diffusion, times of less than 6 minutes, the plot is linear and is characteristic of Case II diffusion in PVC.[9] Due to experimental errors attributable to the more rapid dissolution of PVC in THF, the plot corresponding to Figure 1 showed too much scatter of the data to enable a rate to be determined with sufficient precision to be worth while. Similarly also with CYH, the rapid dissolution prevented assessment.

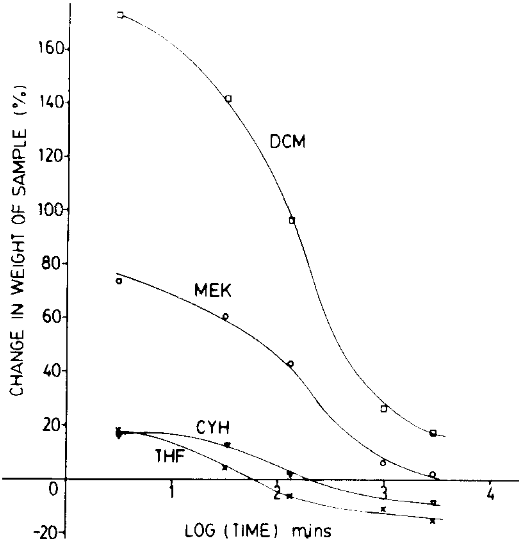


FIGURE 2 Rate of change in weight of sample which have been immersed for one hour in the solvents.

In solvent welding, it is the initial rate of solvent intake (not the diffusion rate at times longer than 6 minutes) which is important since this rate provides some idea of how quickly the mating surfaces may be plasticized and softened in readiness for joining. The initial rates of solvent intake of DCM and MFK could be determined from Figure 1.

The rate of solvent evaporation from the SAZ is as shown in Figure 2. The mass of the samples at the start of the desorption experiments are lowest for specimens in CYH and THF due to the rapid polymer dissolution during the immersion period prior to desorption. The polymer dissolution also caused the weight of the above two specimens to decrease to 9% and 16% respectively below their original weight after desorption for 48 hours. From Figure 2, it is apparent that the samples used in DCM and MEK probably did not dissolve, and that there was residual solvent in the samples even after desorption for 48 hours.

Some of the data from Figure 2 are replotted in Figure 3. From Figure 3, it can be seen that the rate of solvent desorption

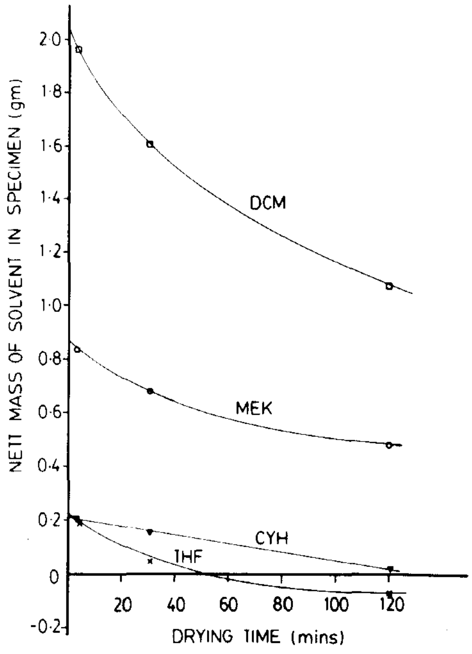


FIGURE 3 Rate of solvent desorption from the SAZ for each solvent (linear plot of results in Figure 2).

decreased with increasing drying time. This probably indicates that the rate of solvent desorption decreases with the solvent concentration in the SAZ. The initial rates of solvent desorption from the SAZ can be determined from Figure 3. The rate of free evaporation of each bonding solvent can be determined from Figure 4. The evaporation rates decreases in the following order: DCM > THF > MEK > CYH. This correlates well with the boiling points of the bonding solvents which are 40°,C, 67°C, 79.6°C and 155.15°C respectively (Table 1).

The diffusion, desorption and evaporation rates determined from Figures 1, 3 and 4 are plotted in Figure 5. No specific trend could be observed when the results were initially plotted against the solubility parameter. However, the diffusion, desorption and evaporation rates all decrease with increasing molar volume of the bonding solvent, the largest molecule having the slowest diffusion rate

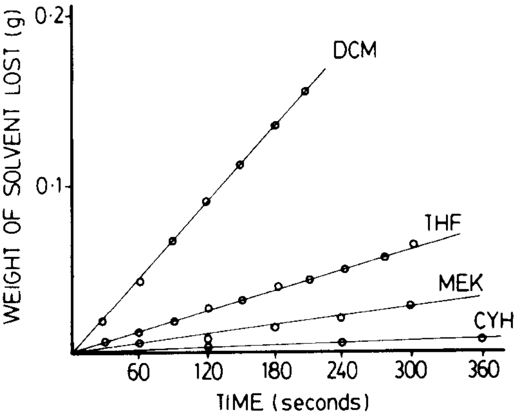


FIGURE 4 Rate of free evaporation of the bonding solvents.

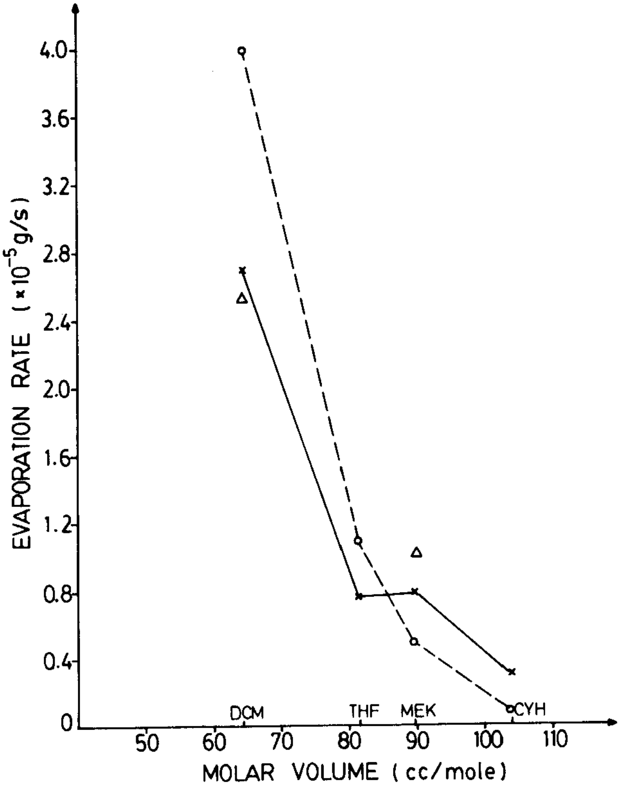


FIGURE 5 Plot of diffusion/evaporation rates against molar volume of solvent (Δ, rate of solvent intake: 0, rate of free evaporation of solvent: x, rate of solvent desorption from SAZ).

(Figure 5). These results are in agreement with Colborn's observations[4] that the molar volume of the solvent is an important factor in considering the solubility characteristics of PVC.

Results in Figure 5 suggest that it would be difficult to obtain a satisfactory solvent weld with DCM since its rate of free evaporation is significantly higher than the rate of solvent diffusion into the PVC. Moreover, with DCM, solvent would rapidly be lost through desorption from an exposed SAZ and this may cause the mating surfaces to lose their tackiness before the surfaces are brought together. Thus a weak and poor solvent weld would be obtained. However it would be easier to solvent weld PVC with MEK since the rate of solvent diffusion into the PVC is higher than the free solvent evaporation rate. With MEK, the mating surfaces are more likely to remain tacky when they are brought together, producing strong solvent welds. The above deduction was confirmed experimentally in that DCM welded joints which had been dried for 24 hours broke when pulled lightly by hand. Examination of the fracture surfaces of the DCM joint (Figure 6) revealed the limited contact between the mating surfaces, producing a weak joint. Strong solvent welded joints were obtained with MEK, THF, and CYH as bonding solvents.

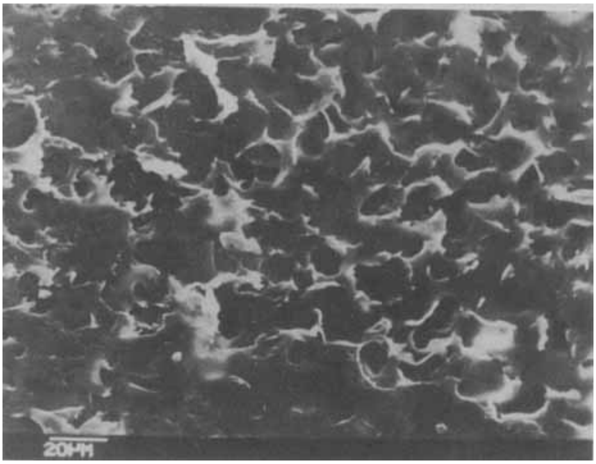


FIGURE 6 Fractograph of a DCM welded joint revealing poor contact at the mating surfaces.

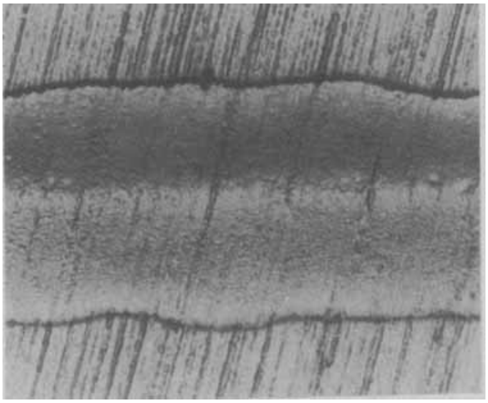


FIGURE 7 Structure of THF solvent weld (magnification 100×).

### Influence of bonding solvents on weld structure

The structure of the solvent welded joints which have been bonded with THF, CYH and MEK are shown in Figures 7, 8 and 9 respectively. Two distinct regions can be seen in Figures 7 and 8. The central band (Figure 7, 8) consist of two SAZs which have been fused together to weld the adherends on the top and bottom of the micrographs. The pair of distinct boundaries in Figures 7 and 8 represent the limit of solvent penetration into the adherends.

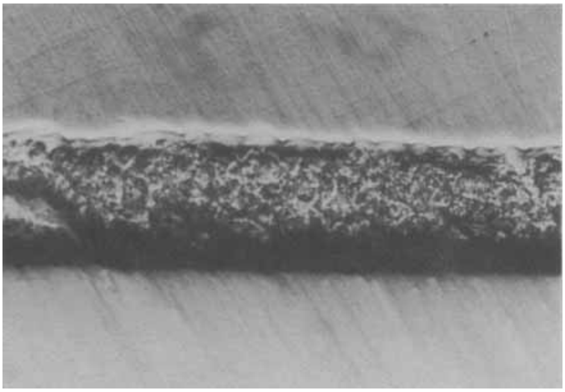


FIGURE 8 Structure of CYH solvent weld (magnification 300×).

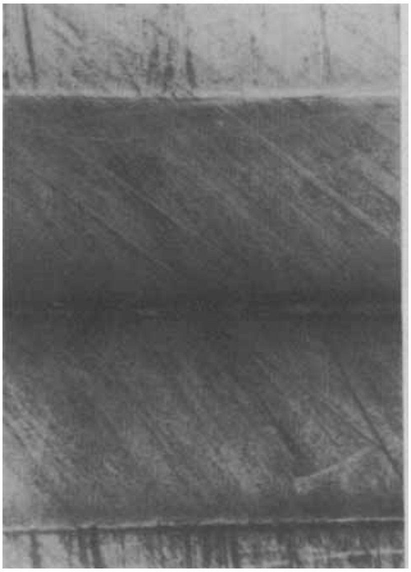


FIGURE 9 Structure of MEK solvent weld (magnification 200×).

The original interface in Figure 7 is located at the light diffused region in the middle of the central SAZ band. Apart for some polishing scratches, it can be seen that good fusion or interdiffusion occurred at the original interface in that no physical boundary exists at the centre of the weld. In contrast, the location of the original interface of the CYH weld (Figure 8) cannot be identified, although the interface is probably at the centre of the weld. There was also good fusion in the CYH welds.

The structure of the MEK welds (Figure 9) is similar to that reported in a previous study[2] (which utilized solvent cements which contained dissolved polymer) although bonding solvents with no dissolved polymer were used in the present work. A distinct and separate 'interfacial layer' was formed at the original interface (middle band in Figure 9). This interfacial layer was sandwiched between the SAZs of the two adherends. As before, the pair of outer boundaries in Figure 9 represent the limit or depth of solvent diffusion into the adherends.

TABLE II
Some experimental results for the solvent welds

| Bonding Solvent | THF | | MEK | | CYH | |
|---|---|---|---|---|---|---|
| Thickness of solvent weld (mm) | 0.95 | 0.15 | 0.50 | 0.10 | 0.05 | 0.02 |
| Microhardness of SAZ ($mm^{-1}$) | 5.4 | | 9.6 | | — | |

Microhardness of PVC = 28.8.

The microhardness of the SAZs of the welds are given in Table II. The microhardness of the CYH weld could not be obtained due to the small width of the SAZ band compared to the size of the indentor of the testing machine. For the THF weld, no difference in microhardness was obtained between a reading taken within the SAZ and another reading taken at the original interface. Hence, the mechanical properties within the SAZ band are probably uniform. The above observation and the fact that no physical boundary exists at the centre of the weld (Figure 7) suggests that there was very good chain interdiffusion across the original interface. The presence of the 'large' diffused region at the centre of the weld (Figure 7) probably indicates that interdiffusion was extensive and was not limited to a small region at the original interface. Interdiffusion can occur because gel layers consisting of chains with high mobility can form on surfaces of adherends which have been attacked by THF, since THF is a good solvent for PVC. The good chain interdiffusion across the original interface suggests there would be no preferencial plane of failure in the THF and CYH welds and this is confirmed by results in the next section.

The microhardness of the SAZ in the MEK weld was higher than that in the THF weld. Although the microhardness of the 'interfacial layer' (Figure 9) could not be determined due to its small width, it was obviously softer than the SAZ. This observation is in agreement with previous work.[9] The presence of the interfacial layer suggests that chain interaction between the adherends was limited to this region. The above two observations probably indicate that the chain network which link the two SAZs was not as extensive as that in the THF and CYH welds. Consequently, failure is expected to occur within this interfacial layer and along the

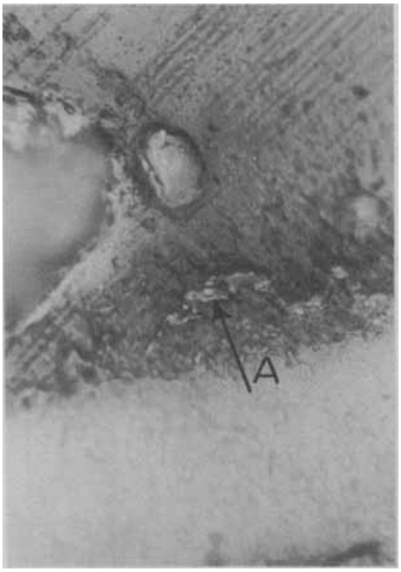


FIGURE 10 Typical crack path in the weld of a necked THF solvent welded joint (magnification 200×).

boundary between the interfacial layer and the SAZ and this is confirmed by results in the following section.

### Failure plane and fractography

Typical crack paths in the necked specimens are as shown in Figures 10, 11 and 12. In the above micrographs, the cracks were propagating from left to right. The fractographs near the edges of specimens which have been tested to failure are given in Figure 13. All the double lap joints which were tested to failure exhibited rapid, brittle-like failure. Despite the different crack propagation rates, the crack paths during the rapid brittle failure and during the traverse of the crack within the weld in necked specimens would be similar since failure always occur along the weakest plane. Hence a study of the crack paths in the necked specimens would help identify the weak planes in the solvent weld and would indicate whether the interchain diffusion across the SAZ resulted in a strong

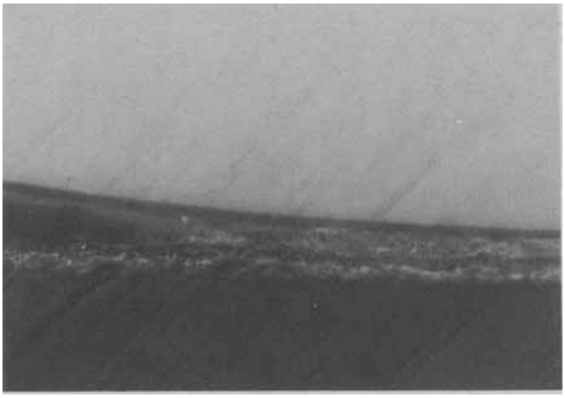
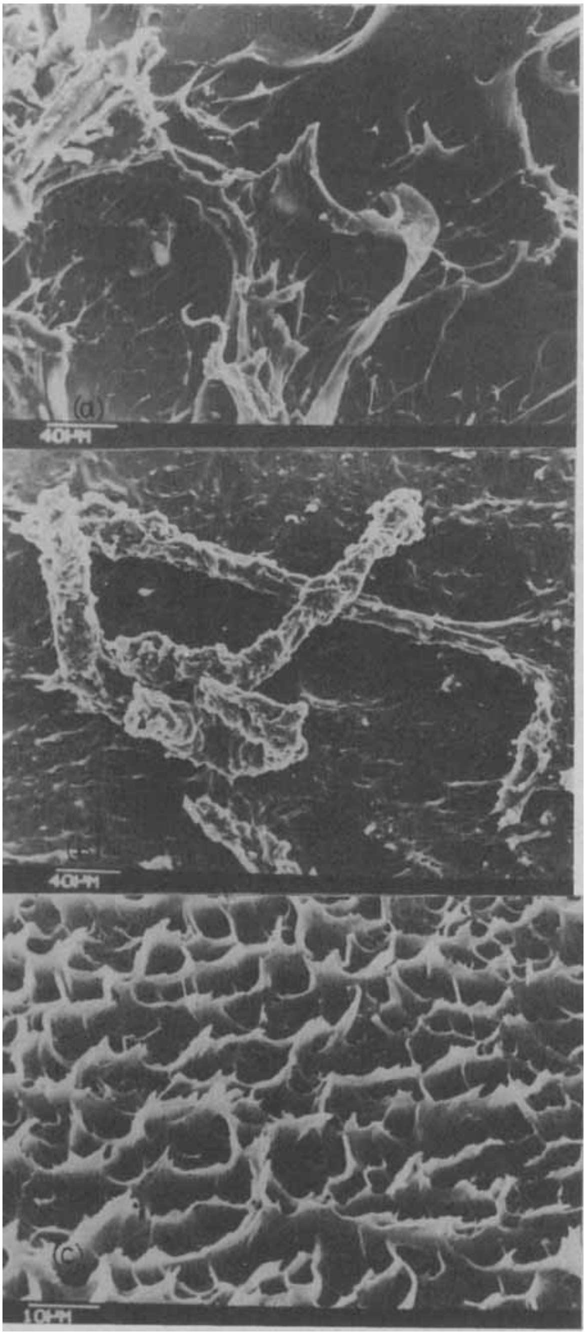


FIGURE 11 Typical crack path in the weld of a necked CYH solvent welded joint (magnification 200×).

FIGURE 12 Typical crack path in the weld of a necked MEK solvent welded joint (magnification 100×).


(a)
40µm
(b)
40µm
(c)
10µm

joint between the adherends. Such a study would also facilitate the understanding of the fractographs.

In Figure 10 the original interface lies within the diffused dark band at the centre of the micrograph. It is clear that the crack did not propagate along the original interface. (The location of the original interface in Figure 10 can be identified by the weld defect (A) in the form of a small region of limited bonding at the mating surface near the bottom right of the blunt crack tip.) The above observation agrees with the prediction in the earlier section and probably confirms that there was very good chain interdiffusion across the original surface. Since there is no plane of weakness in the weld, crack propagation will be accompanied by significant deformation of the matrix at the crack tip. Consequently, the fractograph (Figure 13a) revealed random, irregular and extensive deformation over the entire fracture surface.

It could not be determined if a preferential crack plane existed in the CYH weld (Figure 11) due to the small width of the weld zone. However, the fractograph (Figure 13b) also revealed extensive deformation on the fracture surface. This implies that the chain interdiffusion across the original interface of the CYH welds was also good.

In the MEK weld, the crack propagated preferentially along the interfacial layer (Figure 12) as predicted in the above section. This probably confirms that no extensive chain network linked the SAZ of the adherends. Thus crack advancement would be accompanied only by deformation within the weak interfacial layer which probably has uniform properties. This is why the fractograph (Figure 13c) revealed uniform and regular deformation within the interfacial layer.

Therefore solvents welds with extensive chain interdiffusion across the mating surface and which do not have any preferred plane of failure would have a higher intrinsic strength than solvent welds which have soft weak interfacial layers at the original interface. This issue will be examined in detail in a following paper.

FIGURE 13 (a) Typical fractograph of a THF welded lap joint. (b) Typical fractograph of a CYH welded lap joint. (c) Typical fractograph of a MEK welded lap joint.

## CONCLUSIONS

The most important requirement of a bonding solvent is its ability to dissolve the polymer readily to produce a gel layer of highly mobile chains at the adherend surface. This gel layer will facilitate extensive chain interdiffusion across the SAZ and promote intimate bonding without forming any plane of weakness. Hence there would be no preferred plane of failure. Such solvent welds will lead to high strength since extensive deformation within the weld would accompany both crack initiation and propagation. THF and CYH are good bonding solvents for PVC.

If bonding solvents which are not excellent solvents for the polymer are used, chain interdiffusion between the SAZ would be limited to a small region at the mating surfaces. An interfacial layer which is weaker and softer than the SAZ would then be formed at the original interface. Failure would always occur within the interfacial layer. Such welds would probably have lower strength compared to the former solvent welds since both crack initiation and propagation would only involve deformation within the weak interfacial layer. MEK is a fairly good solvent for PVC.

Other factors such as the solvent diffusion rates in the polymer and in the SAZ are less important. This is because bonding solvents which can diffuse rapidly into the polymer need not be good solvents (*e.g.*, DCM in PVC) and would not form the gel layer of mobile chains which is essential for good bonding.

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