# **The butt-fusion welding of PVDF and its composites**

**Part 2** *The reinforcement and welding of PVDF with short carbon fibres* 

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The effect of reinforcing PVDF with short carbon fibres was studied by injection moulding commercially available and laboratory compounded materials, with the result that components with increased tensile strength and modulus were produced. The extent of the improvements in properties was influenced by the processing techniques used. I n order to ascertain the practical applications of such a material, an investigation of the integrity of butt-fusion welds of the material both to itself and to the unfilled material, was carried out, and on the basis of the findings a procedure for jointing two filled components is proposed. The overall conclusion can be expressed as a strong vindication of the use of both filled and unfilled PVDF as practical engineering materials.

## **1. I ntroduction**

This paper is based on a study of the effect of incorporating short carbon fibres into a PVDF matrix, which then went on to assess the effect of carbon fibres on the welding characteristics of PVDF. The purpose was to see if a significant improvement in mechanical properties could be achieved, bearing in mind that PVDF is already amongst the strongest and stiffest thermoplastics in its unreinforced base state. This study was conducted by injection moulding short carbon fibre filled and unfilled ASTM standard tensile bars which were then tested at standard conditions. A study of various factors which might affect the properties of short fibre reinforced injection mouldings was carried out.

The effects of varying the inherent material properties of a given composite system, such as the fibre weight fraction, the fibre to matrix bond strength, and so on, have been the subject of much discussion in the literature, and to some extent can be explained by a small number of theoretical analyses. However, predictions of the mechanical properties of moulded composites based merely on a consideration of these inherent material characteristics results in poor correlation between theory and experiment. It has become well established that the compounding and moulding operations are highly influential in determining the properties of the final composite system. In this study, therefore, we have investigated to a limited extent both areas of influence; namely, the inherent material characteristics and also the major compounding and moulding variables. In this latter category are included such factors as different injection moulding conditions, principally injection temperature and speed, the use of different injection moulding machines, and the differences between single and double mould gating.

A straight comparison between virgin matrix material and its short carbon fibre reinforced counterpart at varying moulding conditions forms the initial base for the work. Different compounding methods were investigated by a comparison of commercial material with laboratory compounded material using the same matrix, fibre and fibre sizing. The fibre sizing was then changed in order to study the effect of modifying the fibre to matrix bond strength. In Part 1 [1] it was shown that unfilled PVDF

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could be successfully welded using the butt fusion welding technique, and that welds could be produced which were as strong as the base material. This paper extends to the study of the butt fusion welding of carbon fibre filled PVDF, both to itself and to the unfilled matrix material. The reason for undertaking this investigation lies in the fact that there is an increasing interest in fibre filled thermoplastics from the industrial sector, owing to the high specific strengths and stiffnesses of such materials, coupled with their ease of manufacture and fabrication. The most important examples of these two processes are injection moulding and butt fusion welding, respectively. As PVDF is now firmly established as a high performance engineering thermoplastic in its unfilled form, it is natural that a considerable amount of attention has been focussed on ways of improving its specific strength and stiffness. Indeed, this attention has manifested itself in the appearance of commercially available carbon fibre reinforced injection moulded pipe system fittings.

It was the purpose of the presently reported work to investigate the feasibility of incorporating an injection moulded component made from this material into a system which, in practice, is likely to consist predominantly of unfilled components. In order to present a complete feasibility study of such a system, it was considered necessary to investigate the integrity of butt fusion welds between both filled to filled components and filled to unfilled components.

## **2. Experimental details**

## 2.1. Compounding and moulding studies

#### *2. 1.1. Test materials*

The materials used for the injection moulding were:

(a) SOLEF X8N. This is a grade of unreinforced PVDF with a quoted melt flow index of 35 to 70 g  $(10 \text{ min})^{-1}$  at 230°C under 10 g.

(b) A commercial grade of PVDF reinforced with carbon fibres, using matrix material identified in (a), and Grafil XA-S fibres, which have the following properties: tensile strength, 3 GPa, tensile modulus, 230 GPa, specific gravity, 1.82.

(c) Laboratory compounded material of carbon fibres in a PVDF matrix. This is discussed below.

## *2. 1.2. Compounding*

In order to investigate the effect of fibre sizing on the mechanical properties of composites, two separate batches of PVDF filled with carbon fibres which had different sizings were compounded in the laboratory prior to injection moulding. One sizing, polymethyl methacrylate, is the same as that used in the commercial production of carbon fibre filled PVDF. The second batch used fibres coated with an epoxy sizing, which is not normally used with PVDF. The fibres were Grafil XA-S.

The compounding was carried out on a Reifenhauser single screw extruder (30 mm diameter) fitted with a crosshead die. Before the compounding operation began, the screw and die were removed for thorough cleaning, and prior to compounding proper the extruder was purged using regrind PVDF.

The fibres, in the form of a continuous tow, were drawn through the crosshead die in a direction orthogonal to the screw axis and coated with molten polymer emerging from the extruder barrel. The coated fibres were cooled in a water bath and taken up using a variable speed haul off. The thickness of the coating, and hence the volume fraction, could be varied by regulating the haul off speed. The temperatures in the Reifenhauser increased gradually from  $210^{\circ}$ C at the hopper end to  $250^{\circ}$ C at the crosshead die, and a screw speed of 20RPM was used. The coated fibres were then fed into a variable speed pelletising machine which reduced the continuous coated tow to pellets 3mm long. Samples were taken from each batch to be used in determining the fibre weight fraction for the three carbon fibre reinforced PVDF compounds. The procedure used here followed closely that adopted by Folkes [2] of this laboratory for the compounding of long length fibre reinforcement of thermoplastics.



## *2. 1.3. Injection moulding*

The commercial.matrix grade X8N and the three fibre reinforced materials were injection moulded into ASTM standard tensile bars. Two injection moulding machines were used for this work: a Bone Cravens Daniels 350-120 machine fitted with a 45 mm diameter screw, and a Sandretto 6GV/50 machine fitted with a 40mm diameter screw. The injection moulding machine used for



*Figure 1* (a) Fibre lengths for compound A, after moulding into tensile bar. (b) Fibre lengths for compounds B and C (indicated by cross-hatching), after moulding into tensile bars.

the preparation of test specimens is quoted in the appropriate section of the text.

#### *2. 1.4. Fibre length distribution*

Samples taken from injection moulded tensile bars of commercially supplied filled material and laboratory compounded filled material were taken to ascertain the distribution of fibre lengths and the effect of the various processing methods on this distribution. Small samples were placed in a filter paper thimble inside a Soxhlet reflux condensing column and cylcohexanone was refluxed through the column for 10h. The condensed solvent washed away the matrix material leaving the fibres deposited in the bottom of the thimble. This method was used to alleviate the possible fibre breakage which might have occurred due to the turbulent physical effects of the usual methods employing boiling solvent. The fibres were floated off the thimble, washed in acetone and distilled water, dried and transferred carefully to a microscope slide. The fibres were then photographed and the lengths measured by direct comparison with a graticule photographed at the same magnification. Histograms were drawn off the fibre length distributions and are shown in Fig. 1,250 fibres were measured in each batch. These were gained from samples made under conditions most likely to lead to fibre length attrition, that is, the use of the small screw diameter injection moulding machine and the long runner and small diameter gate ASTM bar mould cavity.

Samples of laboratory compounded materials were taken after pelletizing, carefully weighed, and placed in a filter paper thimble inside a Soxhlet

refluxing column as described above. After refluxing, the weight of the fibres was measured and this was compared with the original weight of the fibres coated with matrix material. From this comparison, a figure for the fibre weight fraction was obtained for each compounded material.

## 2.2. Welding trials

#### *2.2. 1. Test specimens*

All specimens, both welded and unwelded, were taken from 6mm by 10cm square injection moulded plaques. In the following discussion, filled specimens will be denoted "F", and unfilled "U", with welded specimens marked by a stroke (/). For example, a filled to unfilled welded bar is described thus F/U.

The investigation took the following form. Tensile specimens were taken from F plaques in directions parallel and orthogonal to the injection direction to see if there was a variation in tensile strength in different directions in the moulding. F/F tensile specimens were then made and the strengths compared with those of the F bars; fibre orientation in the weld region was characterised. F/U bars were then made and tested. The F, F/F and F/U tensile strengths were compared with the results obtained in Part 1 [1] for the tensile strengths of welded and unwelded unfilled pipe systems. Attention was then directed to the flexural mode of testing. The flexural strengths and stiffnesses of rectangular F bars were compared to those of similar F/F bars, and a brief study of the effects of moulding and welding conditions on the flexural properties of the F/F bars was carried out. The response of F/F welds, to the two different modes of testing, tensile and flexural, was compared. Finally F/U/F flexural specimens were made and tested, and the properties of this type of system were compared with those of the F and F/F specimens.

# *2.2.2. Test materials*

The experiments were restricted to two materials: (a) SOLEF  $X8N - an$  unreinforced grade of PVDF.

(b) Grafil XA-S carbon fibre reinforced grade of (a) containing  $10 \,\text{wt}\%$  of fibres, and referred to as Compound C in Section 2.1.2.

The two compounds were injection moulded into the 10 cm square, 6 mm thick plaques at melt temperatures of 220 and  $240^{\circ}$ C, for a fixed mould temperature of  $80^{\circ}$ C, and two injection speeds.

## *2.2.3. Weld procedure*

A Haxey Mk II butt fusion welding machine was used. This machine incorporates on-axis pressure loading, the pressure being applied via a compressed air line. The advantage of on-axis, as opposed to base-loading, is that in base loading there is a movement about the ends of the pipe such that the pressure is not even on all regions of the pipe welding surface. The hot plate is controlled by a thermostat to within  $2^{\circ}$ C. The butt fusion technique for welding thermoplastic materials is as follows:

(i) The faces to be welded are machined flat and parallel.

(ii) The faces are wiped clean and dry.

(iii) The hot plate temperature is checked and the hot plate placed in position between the weld faces.

(iv) The faces are simultaneously brought into contact with the hot plate and a slight pressure is applied (or the order of 0.1 bar). The pressure needed to overcome friction in the welding machine rams, which has been measured before the welding process began, is added to any required pressure. The surfaces are then closely examined to see when the bead begins to form.

(v) When a uniform, small bead has formed the pressure is released and the faces are allowed to remain in contact with the hot plate for a predetermined time. This is known as the soak time.

(vi) On completion of the soak period the hot plate is removed, the faces brought together and the required welding pressure immediately applied. This part of the welding process is completed as

quickly as possible, usually within 3 sec, to try and prevent the weld faces 'cooling and forming a skin. The faces are held together under pressure for a predetermined length of time.

(vii) The pressure is then released and the weld is allowed to cool for 10 min.

Before welding process commences a visual inspection is made of the two components brought together to check for misalignment and mismatch.

For bar to bar welding, specially designed brass clamps were manufactured in order to obtain a high degree of accuracy in the alignment of the two components.

All welds were examined visually after being allowed to cool. In the event of an obviously low integrity weld being formed, for example by allowing the bead to form too large, or by slipping of one or both components in the grips, the weld was cut out and the process repeated. When a visually satisfactory weld had been produced, tensile and flexural stress specimens were cut as required across the weld.

## *2.2.4. Mechanical testing of welded specimens*

Tensile bars were cut from the injection moulded 10 cm square plaques in directions parallel and perpendicular to the injection direction. These specimens were tapered so as to eliminate stress concentration effects at the shoulders of the specimens. The bars were prepared by securing rectangular strips, cut from the plaques, into a jig of the required shape and dimensions. The material was cut to the approximate size using a jewellers coping saw. Each specimen was hand polished using successively finer grades of carborundum paper; grades 180, 220, 400 and 600 were used, and the polishing was finished off with "gamma" alumina paste.

Tensile bars were cut from the bar to bar welded specimens and prepared by hand as described above. Inserts manufactured from polymethyl methacrylate were placed inside the jig so that the bead of the butt weld was not damaged by the surfaces of the jig.

Tensile tests were carried out at controlled room temperature and humidity and at a constant crosshead speed of  $0.5 \text{ cm min}^{-1}$ . For butt fusion welded specimens the cross-sectional area measured was the area of the welded faces prior to welding; that is, the increased area due to the weld bead was not taken into account.

Injection temperature $(^{\circ}C)$	Injection speed	Yield strength (MPa)	Modulus (GPa)	Breaking strain (%)
220	slow	$49.7 \pm 0.4$	$1.2 \pm 0.1$	$101 \pm 37$
220	fast	$49.5 \pm 0.5$	$1.1 \pm 0.05$	$88 \pm 23$
240	slow	$49.6 \pm 0.7$	$1.1 \pm 0.06$	$140 \pm 35$
240	fast	$49.2 \pm 0.9$	$1.1 \pm 0.08$	$110 \pm 29$

TABLE I Tensile properties of unfilled PVDF bars

Butt fusion welds between bars were also tested for strength using flexural mode applied stress. Four or five parallel bars were cut from each welded piece orthogonal to the weld line. In the case of bar to bar welds, the specimens were cut such that the weld bead was situated in the middle of the specimen, measured along the long axis. In the case of bar to bar welds, the bars were cut such that the two welds were equidistant from the centre of the specimen, again measured along the long axis. The sides of the bars were machined to give a smooth surface. Bars were also cut in a similar way from unwelded plaques of filled material for comparison purposes.

In order to examine more fully the integrity of butt fusion welds between combinations of filled and unfilled PVDF, four point bend tests were carried out upon rectangular bars containing F/F and F/U/F welds. Before the welding was carried out unwelded F bars were tested using four point loading to obtain a reference strength and modulus for the welded bars. In this way, a "weld factor" for flexural testing could then be obtained, and compared with the weld factor in tension to see in which loading mode the welds best approximated to the unwelded strength. Four point bending was chosen in preference to the perhaps more usual three point bend test, because the loading anvil in the latter test would interfere with the weld bead in F/F bars. All values quoted in Tables I to VII are the means of six results.

#### **3. Experimental results – compounding and injection moulding**

## 3.1. The effect of varying the moulding **conditions**

Single end gated ASTM standard tensile bars of virgin PVDF were injection moulded on the Daniels machine using four different combinations of injection temperature and injection speed, these being the two variables having the greatest influence on mechanical properties. Two different moulding temperatures were used, 220 and  $240^{\circ}$ C,

coupled with low and high speed injection. The high speed was twice the low speed, and the injection rate corresponding to high speed injection was 100 cm<sup>3</sup> sec<sup>-1</sup>. A mould temperature of  $80^{\circ}$ C was used throughout, with an in-mould cooling time of 60sec. The bars were then stored and tested within 2 to 4 weeks of moulding in accordance with the raw material supplier's suggestions. The bars were then tensile tested at a constant crosshead speed of  $0.5 \text{ cm min}^{-1}$ .

The results are summarized in Table I. The yield strength varies very little with change in moulding conditions; although some variation is apparent, this is within experimental error and cannot be regarded as significant. The figure of 49 MPa is very close to that quoted by the manufacturer and also coincides with the yield strength of specimens cut from unwelded pipe material and discussed in Part 1 [1]. The tensile modulus also shows little variation with moulding conditions, an overall average of 1.1 GPa being recorded. When the strain at fracture is considered, however, two principal points can be noticed. The first is that increasing the injection temperature results in a significant increase in the fracture strain; the second is that at both injection temperatures an increase in the injection speed is accompanied by an increase in the fracture strain. These results can be explained in terms of the microstructural changes which are expected to occur with changes in processing conditions.

Tensile bars of carbon fibre filled PVDF were injection moulded and tested in the same way as for the unfilled bars. A commercial grade containing 10% carbon fibres by weight was used. The composite was moulded using the same set of moulding conditions as for the unfilled material. The results of these tests are shown in Table II. Inspection of this table shows us that the changes in the moulding conditions have very little effect on the tensile properties of the filled material. The tensile strength is around 68 MPa in all cases, the variations being effectively insignificant. The same

Injection temperature $(^{\circ}C)$	Injection speed	Fracture strength (MPa)	Modulus (GPa)	Breaking strain $(\%)$
220	slow	$68 \pm 0.7$	$2.7 \pm 0.3$	$5.6 \pm 0.2$
220	fast	$66 \pm 1.0$	$2.5 \pm 0.15$	$6.9 \pm 0.6$
240	slow	$68 \pm 1.5$	$2.6 \pm 0.25$	$5.9 \pm 0.3$
240	fast	$70 \pm 0.5$	$2.7 \pm 0.2$	$5.7 \pm 0.3$

TABLE II Tensile properties of filled PVDF bars moulded from compound C with the Daniels machine

can be said for the tensile moduli. The fracture strains round off to a value of 6% in all but one case, that at  $220^{\circ}$ C and high injection speed; this has a value of 7%. We do not consider this to be a particularly significant difference, particularly when it is compared with the values obtained for the fracture strain of the unfilled material.

The deformational behaviour of these two materials are illustrated in Fig. 2, which are stressstrain plots for an unfilled and filled bar. The unfilled material behaves in a linear elastic manner up to a stress of 10 MPa, corresponding to a strain of 3%, whereupon yielding of the material begins with a gradual fall off in modulus. At 49 MPa the yield point is reached. The strain at this point is 17%. A neck begins to form which propagates along the bar. The work-softening is arrested at

a strain of 39% and the material then work hardens as the strain increases, before failure finally occurs at a strain of 100%. The deformation of the filled bar is markedly different, as Fig. 2 shows. The behaviour is linear elastic up to 55 MPa (corresponding to a strain of 3%) but with a much higher modulus than for the unfilled material. A slight yielding then occurs before failure, which takes place abruptly at 68 MPa (strain of 6%). The reason for the yielding becomes apparent on inspection of the fracture surface of a filled bar. Fig. 3 shows a scanning electron micrograph which is typical of many observed from different parts of the surface. Most of the fibres protruding from the surface are aligned in the direction of melt injection, or close to it, which coincides with the test axis. Whilst the wetting out of the fibres is



*Figure 2* (a) Stress-strain curve for unfilled injection moulded tensile bar. (b) Stress-strain curve for carbon fibre injection moulded tensile bar.



*Figure 3* High magnification SEM image of compound C fracture surface.

good in the sense that very few bundles were observed, this micrograph shows that it is poor on the individual fibre scale. Around most of the fibres is a gap between the fibre surface and the matrix material, with fibre to matrix bonding being thus only partially effective. Holes can also be observed where a fibre has pulled out of the matrix completely. Both of these effects indicate that the transfer of stress from the matrix to the fibres is not being carried out at a particularly efficient level. As the bond between the fibre sizing (PMMA) and the matrix is known to be good, it would appear that the fibre pullout is principally due to the fact that many of the fibres are very short and below the critical fibre length for this system [3]. An estimate of the lower limit for the critical fibre length was estimated to be

 $240 \mu m$ , by substituting the matrix tensile strength for the fibre to matrix shear strength in the calculation of the critical fibre length. Inspection of the histogram in Fig. lb (compound C), which gives the fibre length distribution taken from a tensile test bar, shows that very few fibres were equal to or greater than this length. As this is very much a lower limit, it may be said that effectively most of the fibres in this system were sub-critical. The state of affairs has also been found to exist in other injection moulded systems, such as glass fibre reinforced nylon 66 and other glass fibre reinforced thermoplastics [4]. The improvement in tensile strength of the filled specimens, when compared with the unfilled, is about 40%. Whilst this is a significantly large enough gain in strength to be useful it is not as large as might be hoped for, when the potential strength based on the rule of mixtures [5] is considered.

It is well established that the complex flow patterns which are a characteristic feature of the injection moulding of both filled and unfilled materials [6] result, when fibres are incorporated into the melt, in a distribution of the orientations of these fibres, relative to a reference axis. The form of this distribution is a function of the processing variables [7] as well as of material properties.

For the system considered here, it appears that the orientation is predominantly, though by no means completely, on axis. This is shown by an inspection of Fig. 4 which is a transmission light micrograph taken from a thin section prepared by an Isomet diamond saw. The plane of the specimen is the same as that of one of the sides of the bar, with the injection direction from left to right.



*Figure 4* Fibre orientation in carbon fibre filled injection moulded bar. Injection direction is from left to right.

## **3.2. The compounding of carbon fibre filled PVDF: the effect of changing the fibre sizing**

The injection moulding of composites, as for unfilled materials, requires the preparation of the composite into a form suitable for use in an injection moulding machine. In order to achieve optimum heterogeneity of the melt, it has been established that the material should be of the form of small pellets, typically 5 mm long or less. This ensures adequate melting of the composite and promotes good dispersion of the fibres throughout the melt. However, it has the disadvantage of setting an immediate upper limit on the length of the fibres, and this limit may be further decreased by degradation of the fibres during the moulding operation itself. The mechanical properties of the finished article will be very highly dependent upon the initial form of the composite as determined by the compounding operation. It is at this stage that such factors as fibre weight fraction can be chosen, and the integrity of the fibre to matrix bond can be influenced by the choice of the fibre sizing. In this section, the tensile properties of two laboratory compounded materials are compared with those of the standard commercial grade used previously and discussed in the previous section. An attempt was made to improve upon the mechanical properties of the standard compound, principally by increasing the fibre weight fraction, but also by increasing the mean fibre length. The importance of choosing a suitable fibre sizing was illustrated by comparing two different sizings in otherwise identically compounded materials.

The two laboratory compounded materials were produced on a Riefenhauser single screw extruder using the "fibre coating" technique employing continuous tows fed through a crosshead die [2]. The continuous composite was then pelletized to give pellets 3 mm long. The fibre weight fraction was determined by solvent washing of the composite and found to be 0.15 in both cases. The three compounds were then moulded on the Sandretto 6GV/50 moulding machine using a melt temperature of  $240^{\circ}$ C and a fast injection speed. Prior to tensile testing, samples were taken from bars of each material and analysed to determine the fibre length distributions.

The tensile fracture strength, tensile modulus, and breaking strain were determined for each material, and fracture surfaces examined in the

TABLE III Tensile properties of filled PVDF bars moulded from compound A, B and C with the Sandretto machine

Material	Fracture strength (MPa)	Modulus	Breaking strain $(\%)$
Compound A	$100 \pm 2.1$	$2.7 \pm 0.2$	$6 \pm 0.2$
Compound B	$92 \pm 4.5$	$2.7 \pm 0.2$	$7 \pm 0.4$
Compound C	$65 \pm 1.9$	$2.9 \pm 0.2$	$5 \pm 0.3$

scanning electron microscope. The results of this testing are shown in Table III.

Two principal comparisons are of interest here. Firstly, the laboratory compound A, a composite of PMMA sized carbon fibres in PVDF, is 54% stronger again than the commercial compound C, which also consists of PMMA sized fibres. Compound A has a tensile strength of 100 MPa, which is twice the yield strength of the unfilled material. The breaking strains and tensile moduli of compounds A and C are effectively the same. Secondly, compound A is 9% stronger than compound B, which consists of epoxy sized carbon fibres in PVDF. The tensile moduli and breaking strains of these two compounds are very similar.

Using the same materials, the laboratory compound A was shown to exhibit a 50% increase in strength over the commercial compound C. This is a significant improvement. An important difference between the two compounds is that A has a weight fraction of fibres of 0.15, compared to 0.1 for C. However, this increase is not sufficient to account for the added strength. The reason for the extra strength can be seen by a comparison of the fibre length distributions of A and C, shown in Figs. la and c, respectively. Compound A, whilst having the same median peak as compound C, has a much broader distribution, with a sizeable fraction of fibres longer than  $500 \mu m$ . These long fibres, not found in compound C, result in a mean fibre length of 590  $\mu$ m for A. This much improved mean fibre length and broader distribution, coupled with the presence of a fraction of supercritical fibres, results in a corresponding increase in the composite's load bearing ability.

Compound B, laboratory compounded using epoxy sized carbon fibres, was 93% as strong as compound A. Epoxy sizings are not normally used when fibres are incorporated into a PVDF matrix, as the PVDF-epoxy bond is not as strong as the PVDF-PMMA bond which exists in A and C. The decrease in strength of this compound,



*Figure 5* High magnification SEM image of compound B fracture surface, showing fibre clumping.

then, is hardly suprising. However, correlation of the information provided by the fibre length distributions and fracture surface micrographs leads us to suggest that the fall in strength occurs largely due to the narrower fibre length distribution which is found for B. This is shown in Fig. lb. Comparison of this with the distributions for A. and C shows that whilst B contains much longer fibres than C, its distribution is not so broad as that for A. The mean fibre length here is  $510 \mu m$ . compared to 590  $\mu$ m for A and 250  $\mu$ m for C. The weight fractions of A and B are the same.

Inspection of fracture surface micrographs of all three compounds show that wetting out is generally poor, and worst in the case of compound B, as expected. In the latter case, not only is individual fibre wetting out poor, but there was also evidence of fibre clumping as shown in Fig. 5. This compound would be particularly prone to fibre pullout were it not for the existence of long fibres which can carry larger loads. The matrices in the three compounds all appear to have behaved in a ductile manner prior to failure. The holes in the matrix, visible on all fracture surfaces, indicate that failure has been initiated by the pulling out of short fibres, although there is some evidence of fibre fracture also, for example as seen in Fig. 3. In composites of this type, in which such a sizeable fraction of the fibres are sub-critical, this is to be expected.

#### **3.3. The effect of double end gating**

The tensile bars discussed in the above sections

were moulded in a twin bar mould, both individual bars being gated at one end each. In practice, however, injection moulded components are invariably made in a mould cavity into which the polymer melt enters through more than one gate. Also, there is often a protrusion such as a pin into the cavity, around which the melt front diverges before reforming on the other side. In both of these cases, a situation arises where two or more flow fronts meet, and an internal weld is formed. Depending upon the way in which the component is subsequently loaded, this internal weld may act as a weakened region, and lead to premature failure of the component. The intersection of an internal weld line with an external butt fusion weld bead, such as occurs at welds between injection moulded fittings and extruded pipe, has been identified as a potential failure initiator under fatigue loading [8]. In order to investigate the integrity of internal welds in both filled and unfilled materials, one half of the mould was modified such that one of the two bar cavities was gated at each end. The other bar cavity was left with a single end gating system so that a direct comparison between the two forms of gating could be made.

The four materials used in this experiment were:

(i) unfilled PVDF (matrix material)

(ii) commercial carbon fibre filled PVDF (compound C)

(iii) laboratory compounded carbon fibre filled PVDF, using a PMMA sizing (compound A)

(iv) laboratory compounded carbon fibre filled PVDF, using an epoxy sizing (compound B).

For each material and cavity design a mean tensile strength, tensile modulus and breaking strain was determined. The results of this investigation are summarized in Table IV.

#### *3.3. 1. Unfilled bars*

Inspection of Table IV shows that for unfilled PVDF bars the introduction of a double end gate does not adversely affect the tensile yield strength or modulus. The strengths and moduli of the bars containing an internal weld line are exactly the same (to within experimental scatter, which was of the order of 1%) as the strengths and moduli of the single-gated gars. The strains to break, however, are significantly less; the double-gated bars have a breaking strain of around 40% that of the single gated bars. In some cases, the double-gated

Material	Mould	Tensile	Tensile	<b>Breaking</b>
		strength (MPa)	modulus (GPa)	strain $(\%)$
Virgin PVDF	<b>SEG</b>	$50 \pm 0.5$	$1.2 \pm 0.1$	$101 \pm 37$
Virgin PVDF	<b>DEG</b>	$50 \pm 0.5$	$1.2 \pm 0.1$	$37 \pm 4$
Compound A	<b>SEG</b>	$100 \pm 2.1$	$2.9 \pm 0.2$	$6 \pm 0.2$
Compound A	DEG.	$88 \pm 3.1$	$2.7 \pm 0.2$	$5 \pm 0.3$
Compound B	<b>SEG</b>	$92 \pm 4.5$	$2.7 \pm 0.2$	$7 \pm 0.4$
Compound B	DEG	$81 \pm 2.9$	$2.6 \pm 0.3$	$5 \pm 0.4$
Compound C	<b>SEG</b>	$65 \pm 1.9$	$2.9 \pm 0.2$	$5 \pm 0.3$
Compound C	<b>DEG</b>	$61 \pm 1.8$	$2.8 \pm 0.3$	$4 \pm 0.3$

TAB LE I V The effect of multiple gating on tensile properties. SEG and DEG indicate single end and double end gating, respectively

bars deformed in a similar manner to the singlegated bars. In these cases, the bar did not fail in the vicinity of the internal weld, but failed after necking elsewhere in the gauge region of the bar. The weld clearly had no effect on the mechanical properties in these cases and could not be described as a weak point. However, most bars failed by crack propagation through the bar at the weld line. A stress-strain curve for a failure at the weld is shown in Fig. 6. The deformational behaviour is initially very similar to that of a single-gated specimen. The principal difference



*Figure 6* Stress-strain curve for unfilled double end gated injection moulded tensile bar.

lies in the abrupt failure at just under 40%, after some yielding but before the onset of necking. Comparison of Figs. 2 and 6 shows that the work softening region ends at an identical strain in the two types of specimen. Visual inspection of the internal weld region shows a very shallow line running around the bar and orthogonal to the axis at all points. Microtomed specimens taken across this weld region reveal no microstructural changes across the weld for this set of moulding conditions; the line is confined to the surface of the bar. In the majority of cases, where failure occurs at the weld, this line will act as a microcrack and initiate failure. However, the integrity of the weld is such that yielding can occur before the crack propagation becomes catastrophic.

#### *3.3.2. Filled bars*

The influence of double-gating on the tensile properties of carbon fibre reinforced PVDF is much more marked than for the unfilled material, as is apparent from an inspection of Table IV, which summarizes the mechanical properties of the three different filled materials used. It should be pointed out that this comparative set was carried out using bars moulded on the Sandretto moulding machine, which gave slightly lower strengths than the Daniels machine. The difference in strength, whilst lying well outside experimental scatter, was not particularly large on an absolute scale. The principal reason for the strength difference [9] would appear to be increased fibre degradation owing to the use of a smaller screw with a shorter inter-flight distance.

The commercial material, compound C, was also moulded on the Daniels moulding machine, for a direct comparison with the unfilled material; but the ratios of strengths, strains and moduli of double gated to single gated mouldings were the



*Figure* 7 (a) Fibre orientation at the internal weld in a double end gated filled injection moulded tensile bar. (b) Fibre orientation away from the internal weld in double end gated filled injection moulded filled tensile bar.

same on both machines. Table IV hence quotes the values obtained from Sandretto mouldings, and a direct comparison can then be made with laboratory compounds A and B. A distinct loss in tensile strength occurs for all three filled materials due to the introduction of double end gating. The two laboratory compounds, A and B, lose 12% of their fracture strength, whereas compound C loses 6% of its strength. As the commercial compound is much weaker than the laboratory compounded materials, it is not surprising that this system, which is less efficient in reinforcement; is affected the least. Fig. 7a shows the fibre orientation in the internal weld region. The plane of the micrograph is the same as the plane of one of the sides of the bar, with injection direction from left to right. This micrograph can be compared with Fig. 7b which shows a region in the same plane, but away from the weld region. It can clearly be seen that in the vicinity of the internal weld the fibre orientation is predominantly in a direction orthogonal to the injection direction, whereas away from the weld region (Fig. 7b) the fibres are largely aligned along the injection direction. These micrographs are taken from bars moulded using compound C. There is a considerable angular distribution of fibres at the edges of the weld region, so that there exists a finite volume of non-aligned and hence only slightly reinforced material. This is reflected in the location of the fracture planes in individual bars. The distance from a marked point on the bar to the centre of the fracture surface was measured for each individual bar, and it was found that this distance varied by up to 1.5 mm. This can be taken as the effective width of the integral weld region. It should be noted that this concept of an affected volume is in contrast to the unfilled material situation, where failure, if it occurred at the weld, always took place along a very well defined plane, with no variation in position lengthwise along the specimen.

The deformational behaviour of the singleand double-gated filled bars is shown in the stressstrain curves for each of the three compounds (Figs. 2, 6 and 8). For each compound, changing from a single to a double gate results in three principal effects:

- (i) the fracture stress decreases;
- (ii) the fracture strain decreases slightly;

(iii) less yielding occurs, and the specimens fails in a more brittle manner.

The tensile modulus, which measures the material's initial response to the applied load, is hardly affected at all by the change in gating system.

At the particular set of moulding conditions used, unfilled PVDF is not weakened by the presence of an internal weld line in the moulding. However, the use of such a moulding may be restricted by the fact that its strain at failure is severely reduced. For filled bars, the picture is different. The fracture stress is reduced by a significant percentage, as fibres within the weld region are aligned orthogonal to the flow direction, which coincides with the test axis.

#### 3.4. The effect of ageing and strain rate on tensile properties

The embrittlement of carbon fibre reinforced composites with age is a well known problem. To investigate whether or not this was a problem in



*Figure 8* Stress-strain curves for double end gated injection moulded filled bars: (a) compound A; (b) compound B; (c) compound C.

carbon fibre reinforced PVDF, injection moulded bars of both the unfilled matrix material and filled commercial grade, compound C, were stored in the dark in sealed containers for seven months prior to testing. They were then tested under identical conditions to those used for newly-moulded bars, which have been described previously. A slight increase in strength was recorded for both the filled and unfilled bars. The moduli and fracture strains were not affected. Whilst the strength increases were outside experimental scatter, they were not large enough to promote any reevaluation of the behaviour of either the filled or the unfilled materials.

A series of tensile tests was also carried out on injection moulded bars, the strain rate being changed between each test. Compound C was used



STRAIN RATE (cm min<sup>-1</sup>)

*Figure 9* The influence of strain rate on the tensile strength of a filled bar, single end and double end gated, respectively.

for this test, and the strain rate was varied between  $0.05 \text{ cm min}^{-1}$ , and  $2.5 \text{ cm min}^{-1}$ , which is five times faster than the standard testing rate used previously. The results are presented graphically in Fig. 9 which shows the value of the tensile fracture stress at each of the strain rates used. A steady increase in strength with increasing strain rate can clearly be seen, for both single and double gated bars. This is in agreement with results found to be generally true for unfilled materials.

#### **4. Experimental results - welded bars** 4.1. The tensile strengths of F bars

Tapered tensile bars were cut from F plaques, in directions parallel and orthogonal to the injection direction. They were then hand polished before tensile testing at a constant crosshead speed of  $0.5$  cm min<sup>-1</sup>. Tapered tensile specimens were used for two reasons:

(i) preliminary- testing of dumb-bell shaped specimens always resulted in failures at one of the shoulders at the end of the gauge region.

(ii) tapering the specimens had the effect, in the case of the welded specimens, of concentrating

TABLE V The tensile strengths of tapered bars

Bar type (see text)	Tensile strength (MPa)	
U	$57 \pm 2$	
U/U	$53 \pm 2$	
F/U	$53 \pm 2$	
F	$85 \pm 4$	
(parallel to injection direction)		
F	$89 \pm 4$	
(orthogonal to injection direction)		
$_{\rm F/F}$	$57 \pm 2$	

the load at the weld region, and this aided the identification of failure regions in the weld.

The results of this testing are summarized in Table V. The bars cut from a direction orthogonal to the injection direction are slightly stronger than those cut parallel to this direction. Whilst the difference in strength is large enough to be of note, it should be pointed out that it is the same size (6%) as the experimental scatter for both types of specimen. However, a higher strength for the specimens orthogonal to the injection direction is to be expected when the fibre orientation in the original plaque is considered. Fig. 10 shows an optical micrograph of a thin diamond saw cut sample taken normal to the injection direction. It can be seen that a large fraction of the fibres lies in this plane, transverse to the injection direction. A tensile specimen taken in the region of this plane, with its long axis parallel to the plane, will thus be more efficiently reinforced than specimens taken orthogonal to the plane. Micrographs



*Figure 11* Fracture surface of filled tapered tensile bar, bar axis parallel to injection direction.

of typical fracture surface of both types of specimen are shown in Figs. 11 and 12. Both fracture surfaces appear similar at low magnification and it is only when the surfaces are observed at fibre dimensions that any differences are noticeable. The principal difference lies in the state of the matrix, which is slightly more ductile in the parallel-direction bar. In both cases, there is evidence of fibre pullout and apparent fibre fracture. The wetting out of individual fibres bundling was found, indicating good dispersion in the melt.

A comparison of the mean tensile strengths of bars cut from the 6 mm thick injection moulded plaques with the strengths of the injection moulded bars, as discussed in Section 3, shows that the thicker plaque bars are 25% stronger on average than the thin injection moulded test bars. This could be partly accounted for by less fibre length attrition in the moulding of the 6 mm thick



*Figure 10* Fibre orientation in filled injection moulded plaque, where the plane of the section is normal to the injection direction.



*Figure 12* Fracture surface of filled tapered tensile bar, bar axis orthogonal to injection direction.

plaques, where the mould cavity had a large diameter gate and a short runner, and was moulded on the large diameter screw injection moulding machine.

## 4.2. The tensile strengths of F/F bars

F plaques were cut along a plane halfway down the plaque measured in the injection direction, and orthogonal to this direction, and then butt fusion welded back together again. A welding temperature of  $205^{\circ}$ C and a welding pressure of 0.3 bar were used, these being optimum welding conditions obtained from the data in Part 1 [1]. Four tapered tensile specimens were prepared from each welded plaque such that the weld region was located at the midpoint of the long axis of the specimen. The bars were tested at identical conditions to the F bars discussed above.

The mean tensile strength of these bars was 57 MPa. This gives a weld factor, when compared with the strengths of the F bars, of 0.66, which is low when compared with the weld factors of 1.0 which can be obtained in welds of unfilled PVDF. In general, this figure would be regarded as unacceptable, unless the welded system was to be subjected to very low loads, in which case filled components would be unnecessary. Also, the presence of a weld of this standard reduces the strength increase of the system, when compared to an unfilled system, to only 16%. The reason for this poor performance becomes apparent when the fibre orientation in the weld region is examined [10].

Fig. 13 shows a diamond saw cut specimen taken across the weld region, such that the direction of the applied load is across the plane of the micrograph. It can be seen that to either side



*Figure 13* Fibre orientation in an F/F butt-fusion weld.



*Figure 14* Typical fracture surface of an F/F butt-fusion weld.

of the weld region the fibres are predominantly aligned along the tensile test axis, giving good reinforcement, whereas the fibres in the weld are aligned orthogonal to the loading axis and hence are not acting as a reinforcement in this region. The flow of the fibres towards and into the weld bead is clearly evident in this micrograph, and it is interesting to compare it with Figs. 7a and b, which shows the fibre orientation in an internal weld. The off-axis orientation in the butt fusion weld is much more pronounced and the alignment orthogonal to the test axis more regular. This explains why the strength of the external butt fusion weld is much lower than that of the internal weld. The fracture surface of a typical F/F weld is shown in Fig. I4. The specimen has failed down the central weld plane. The micrograph corroborates the information derived from Fig. 13, which implied that most of the fibres in the weld region lie in this plane, and very few protrude outwards, This is in contrast to the two F bar fracture surfaces shown in Figs. 11 and 12, in which most of the fibres protrude outwards from the surface, with a very small number lying in the fracture surface plane.

## 4.3. The tensile strengths of F/U bars

The possibility of incorporating a filled component into a system consisting predominantly of unfilled artefacts leads directly to an investigation into the feasibility of producing highintegrity F/U butt fusion welds. F and U plaques were cut along the same planes as the F plaques in the previous section, butt fusion welded, and tapered F/U tensile specimens prepared in the



*Figure 15* (a) Optical micrograph of thin section taken across an F/U butt-fusion weld. The fibre reinforced side is on the left. (b) High magnification micrograph showing more clearly the difference in microstructure across the centre-line of the fusion weld.

same manner as before. Care had to be taken, during the welding process, to choose welding conditions which were suitable such that adequate melt flow was possible on the F side without causing excessive flow on the U side. The melt flow indices for the two materials, measured in the laboratory prior to welding, differed by a factor of 2.4. This difference did not particularly hinder the welding operation, a finding which is in agreement with the results of de Courcy and Atkinson [11], who conducted an extensive survey into the butt fusion welding of pipes having different melt flow indices.

The mean tensile strengths of the U and F/U bars was 57MPa and 53MPa, respectively. The U/U and F/U bars thus have the same tensile strength, and each have a weld factor of 0.93 when compared with the U bars. Two points are of particular interest:

(i) the reinforcing effect of the fibres is cancelled out by introducing a weld into the system;

(ii) the F/U weld is slightly lower in strength than the F/F weld. The consequences of these two points is discussed below.

First, it is of interest to examine the structural features of the F/U weld. Diamond saw cut specimens, approximately  $50 \mu m$  thick, were taken across the weld in a similar plane to the previous weld investigations. Optical micrographs of the weld are given in Figs. 15a and b. Fig. 15a, taken at low magnification, clearly shows the extent of the weld region and the structural features of the F and U regions on either side (the F region is to the left of the weld). Fibres can be seen in the plane of the specimen on the F side, and a small number can be seen inside the weld itself. The material on the U side is of the same spherulitic form as that found in the pipe specimens discussed in Part 1 [1]. At the edges of the weld the fibres change orientation as the material is sheared upwards towards the external surfaces, and into the bead. Inside the weld region itself the fibres are aligned orthogonal to the direction of load (as in the F/F bars discussed in the previous section), and it is interesting to note that there is no bridging of fibres across the weld between the F and U regions. Fig. 15b, which shows the weld region itself in more detail, confirms this view. An apparent difference in microstructure between the F and the U regions can be seen in both Fig. 15a (which shows the structure away from the weld) and Fig. 15b (which shows the structure inside the weld). The U side appears to consist of a larger scale spherulitic structure both inside and outside the weld than the F side. A clear dividing line running down the centre of the weld can be identified in Fig. 15b, which also shows more fibres aligned orthogonal to the applied load direction. Shear bands, equivalent to zone 4 in pipe to pipe welds, Part 1 [1] can be seen at the outer edges of the weld region.

Visual inspection of the specimens after testing indicated that failure occurred at or near the weld, on the U side. The U bead was invariably broken off at the moment of failure, which happened abruptly. A large area of the F side appeared to be coated with U material, and this was confirmed upon examination of the two fracture surfaces formed for each specimen. Scanning electron micrographs from selected areas of these



*Figure 16* (a) Unfilled side of F/U bar: ductile to brittle transition on a fracture surface. (b) Unfilled side of F/U bar: imprint of short fibre on fracture surface.

are shown in Figs. 16a and b. A comparison of the F and U surfaces, respectively, results in the identification of a large centralized area of ductile material which corresponds, on the F side, to the layer of U material visible. The ductile material is surrounded by a brittle region, with a sharp transition between the two regions, as illustrated in Fig. 16a. This micrograph is taken from the U side. Just inside the brittle region patched surface structure, a characteristic of crazing, can be identified. Patch structure is also visible in Fig. 16b, also taken from the U side, and showing the imprint of a very short fibre which presumably was pulled out of the matrix at failure. Equivalent micrographs of the F side show similar features, and Fig. 17 shows fibres lying in the fracture surface plane. A detailed examination of the F side fracture surface showed that virtually no fibres were



*Figure 17* Filled side of F/U bar: fibres in the fracture surface plane.

protruding from the surface, a result predicted from an examination of the fibre orientation prior to testing, as shown in Fig. 15b.

This investigation of the weld region before and after testing can be summarized in the following way. Before testing, fibres in the F side away from the weld region are aligned sufficiently well along the axis of the applied load to act as an effective reinforcement of the matrix. Near and in the weld region, however, material flow during welding causes the fibres to align orthogonal to the load direction, thus acting as a very poor reinforcement at this point. Effectively no fibres bridge the weld from the F to the U region. Failure occurs on the U side of the weld close to the outer periphery of the weld region on this side. Regions of ductile and brittle material on the fracture surfaces, with a well defined transition between the two, suggests that failure occurs in two stages, a slow deformation followed by catastrophic crack propagation.

#### 4.4. The flexural properties of F bars

In order to examine more fully the integrity of butt fusion welds between combinations of filled and unfilled PVDF, four point bend tests were carried out upon rectangular bars containing F/F and F/U/F welds. Before the welding was carried out unwelded F bars were tested using four point loading to obtain a reference strength and modulus for the welded bars. In this way, a "weld factor" for flexural testing could then be obtained, and compared with the weld factor in tension to see in which loading mode the welds best approximated to the unwelded strength. Four point

bending was chosen in preference to the perhaps more usual three point bend test, because the loading anvil in the latter test would interfere with the weld bead in F/F bars. The flexural strength and modulus were measured to be 250MPa and 3GPa, respectively. Unfortunately, it was not possible to compare the flexural strengths with those of U or U/U bars, as both of these deformed in the testing rig to such an extent that the bars came into contact with the rig itself, and the tests had to be abandoned. However the flexural strength of PVDF is quoted in the commercial literature as being equal to 55 MPa, and this can be used as a comparison with the values for the F bar quoted above. It is clear that the presence of the carbon fibres greatly increases the strength in flexure of the material, the strength increase being more marked than that for the tensile case.

## **4.5. The flexural properties of** F/F bars

F plaques were cut in half along a plane midway along the injection direction and butt fusion welded back together again, as in the preparation of F/F tensile bars. Four rectangular bars were cut with their long axes parallel to the injection direction, such that the weld bead was exactly midway along each bar. After preparation the bars were tested in the same manner as the F bars discussed above. The welding'was carried out at three different sets of welding conditions, in order to observe the effect upon flexural properties that this might have. The welding conditions used were:

(i) welding temperature  $195^{\circ}$  C, welding pressure 0.1 bar;

(ii) welding temperature  $215^{\circ}$  C, welding pressure 0.6 bar;

(iii) welding temperature  $245^{\circ}$  C, welding pressure 1.2 bar.

Also, F plaques moulded at four different sets of moulding conditions were used. Whilst some variation between plaques was found, this was always within experimental scatter, and could be considered negligible.

The results are presented in Table VI, and show that a distinct improvement in the flexural strength can be gained as the welding temperature and pressure increase. However, even the highest mean strength attained, 131MPa, represents a weld factor of only 0.52 when compared with the mean strength of the F bars. It is possible that increasing the welding temperature further

TABLE VI The effect of welding conditions on the flexural properties of F/F bars

Welding temperature $(^\circ C)$	Welding pressure (bar)	Flexural strength (MPa)
Weld free plaque	250	
195	0.1	$94 \pm 10$
215	0.6	$105 \pm 15.3$
245	1.2	$131 \pm 11.6$

could promote better flow of the F material, but it is clear that the over-riding factor is the alignment of the fibres in the welding surface plane, and that unless this problem is overcome, refinements of such factors as welding conditions will be comparatively ineffectual. The commercial hot plate used was working near its upper limit at  $245^{\circ}$  C, and also PVDF begins to degrade at  $280^\circ$  C, so increasing the welding temperature is probably not a viable option in any case.

## 4.6. The flexural properties of F/U/F bars The rationale behind producing F/U/F bars for testing was twofold:

(i) the good weld factor and improved ductility of the F/U welded tensile specimens promoted interest in the behaviour of the F/U weld under flexure;

(ii) the brittleness of the F/F welds, especially in tension, coupled with comparatively low weld factors, pointed to a need for a new approach in the jointing of two filled materials. Whilst other jointing methods can be used for fibre reinforced thermoplastics, butt fusion welding has been so successful elsewhere that it was hoped that its applicability to all types of thermoplastic systems could be demonstrated.

The logical way of investigating both of these ideas was to make a "double" weld by welding a thin (1 cm wide) bar of U material to two larger bars of F material, one on each side. This then gives the F/U/F configuration, and has the advantage of simultaneously providing an F/U weld for flexural testing with a new method of jointing two F components. The width of the thin U bar was made as small as possible, and the actual size was determined by practical considerations; bars with a width of less than 1 cm could not be clamped successfully in the special clamps designed for bar to bar welding. After welding of the bars, flexural specimens were cut and polished as described above, and tested in the four point

$\mu$ exulat properties of $\Gamma$ / $\sigma$ / $\Gamma$ bars			
Welding temperature $(^{\circ}C)$	Welding pressure (bar)	Flexural strength (MPa)	
195	0.1	$121 \pm 15.0$	
215	0.6	$157 \pm 26.0$	
245	1.2	$178 \pm 25.0$	

TABLE VII The effect of welding conditions on the  $f$  and properties of  $E/H/E$  bars

bend rig in the same manner as the for F and the F/F bars. The systematic approach used to produce different types of F/F bars was followed in this investigation; different welding conditions were used, and F plaques moulded at four different sets of moulding conditions were also tried. Also, a small number of F/U/F samples were made by taking the thin U bar from two different' positions in the original U plaque; this was to test for any effects due to molecular orientation in the U bar. The bars were cut either parallel to, or orthogonal to the injection direction.

The results for the flexural strengths of F/U/F bars are shown in Table VII. Similar trends to the variation in properties with welding conditions for the F/F bars are apparent; the flexural strength increases with incresing welding temperature and pressure. In the case of the weld produced at  $245^{\circ}$  C, the welding soak time was reduced from the normal 30 to 15 sec, as preliminary welding showed that a 30 sec soak time resulted in an overlarge bead on the U side.

The results, when compared with those for the F/F bars given in Table VI, clearly show the superiority of the F/U/F bars in respect of the flexural strength. The lowest mean F/U/F strength, 121 MPa, is almost as high as the best F/F strength, 131 MPa. The highest mean strength recorded in this experiment, 178MPa, is equivalent to a weld factor of 0.71 when compared with the strengths of the F bars. (The highest individual strength, 203 MPa, is equivalent to a weld factor of 0.81. This bar was tested in the comparison of F/U/F bars made using F plaques which had been moulded at different moulding conditions. The variations in strength of these specimens was small, and the overall mean strength was 181 MPa, with any variations between plaques lying well inside experimental scatter). These results are of the same order as the F/U weld factors in tension, and consolidate the integrity of the F/U weld.

## **5. Concluding remarks**

A systematic study of the butt-fusion welding of PVDF and a short carbon fibre/PVDF composite has been carried out, as well as an investigation into the compounding and injection moulding of several short carbon fibre/PVDF composites. PVDF has been shown to be a material which can be welded to produce high integrity joints (Part 1) which can be equal in strength to the unwelded material. Within limits, the welding conditions are not critical, and a "window" of suitable welding conditions has been identified. The microstructural features of a PVDF butt-fusion weld have been characterized and found to be essentially similar to those found in welds made with other semicrystalline thermoplastics, with the exception of a phase transformation which occurs inside the weld region, and is perculiar to PVDF.

After confirmation of the viability of using a PVDF jointed pipe system, attention turned to ways of further improving the mechanical properties of PVDF. The results of a comparative study of the injection moulding of commercially available short carbon fibre/PVDF composite and laboratory compounded materials have been reported, and shows that with a judicious choice of materials and processing techniques a composite with mechanical properties greatly superior to those of unfilled material could be successfully produced. The influence on mechanical properties of various factors, including both inherent material properties and aspects of the processing techniques used, was quantified and discussed. In particular, it was shown that under aggressive processing conditions it was possible to routinely produce composite mouldings with internal Weld lines exhibiting a tensile strength of 88 MPa, compared with the unreinforced PVDF strength by the same processing route of 50MPa, but with the expected substantial decrease in strain to fracture.

The high quality of the PVDF pipe system buttfusion welds reported in Part 1, coupled with the excellent mechanical properties of the carbon fibre/PVDF composite, prompted a survey of the butt-fusion welding of the composite, both to itself and to unfilled material. This was influenced by an increasing industrially based interest in carbon fibre filled PVDF, manifested by the appearance of commercially available carbon fibre filled PVDF components. It was found that welding the composite to itself was not successful, owing principally to off-axis alignment of fibres

in the weld, and that welding the composite to unfilled material resulted in similar weld strengths. These strengths were quantified in both tension and flexure, and a way of jointing filled to filled materials was suggested by implanting a thin bar of unfilled material between two filled components. The average elongation to break of a weld between fibre filled and unfilled PVDF, even in the constrained tapered test specimens, was four to five times greater than that of the average between two filled bars. A carbon fibre reinforced part, or an assembly of fibre reinforced parts jointed together by implanting a thin unreinforced section, could be incorporated into a butt-welded system of unreinforced parts, whilst maintaining the same strains and stresses to failure as measured for the joint between butt-welded unreinforced parts. The high stiffness of reinforced parts can therefore be utilized for valves and other functions requireing high stiffness, and the integrity of the PVDF system maintained. The measured high strength of the carbon fibre reinforced material can only be maintained in a system of parts by profiling the ends to be joined to give a larger cross-section weld, or by the use of spigot and socket system of jointing or other modes of reinforcement external to the weld.

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