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Low temperature tensile lap-shear testing of adhesively bonded polyethylene pipe

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Abstract

This work studies the lap-shear strength performance of polyethylene pipeline bonded with acrylic adhesive in the temperature range -10 to +20°C. Single lap shear test samples were firstly prepared at 20°C under various clamping pressures and curing times to determine suitable conditions under which to prepare and test further samples at temperatures of -10, -5, 0, +5 and +20°C. It was found that a decrease in curing/testing temperature to zero degrees resulted in a steady reduction in the lap-shear strength performance of the bonded joints from a mean value of 2.72 MPa at +20°C to 1.15 MPa at 0°C. Below zero degrees the strength of the bonded substrates was significantly reduced; no samples bonded at -5°C had sufficient strength to test and only one sample bonded -10°C was tested, which had very low strength of 0.105 MPa.

Keywords: Polyethylene; adhesively bonded; low temperature; tensile lap-shear; electrofusion welding

1. Introduction

Polyethylene (PE) is well known for its widespread use in the manufacture of natural gas pipelines and fittings in the form of medium-density polyethylene (MDPE) [1], as well as more recently for the production of safety critical nuclear water pipe [2]. PE offers many advantages over traditional metal pipes, such as lower cost, higher strength-to-weight ratio, ease of jointing, higher impact strength, higher flexibility and higher chemical and corrosion resistance [3-7]. However, one key disadvantage of using PE in these applications is the difficulty in producing safe and reliable pressure tight joints when joining the material together to connect pipelines or services and fittings. This safety critical area is receiving increasing attention as any failure in these pipeline systems invariably leads to significant economic losses as well as potential loss of lives [8].

There are several established techniques for joining of PE pipeline such as fusion welding, friction welding and mechanical joints, but by far the most popular method is electrofusion (EF) welding [9-12]. EF welding usually entails fitting two PE pipes inside a PE fitting or coupler and then melting them together by passing a high current through a high resistance wire element contained within the fitting, see Fig 1 [8]. The high current causes the wire to heat up, which in turn melts the surface of the PE pipes and fitting causing them to fuse together. The fusion process usually takes 24 to 90 seconds to provide acceptable joint strength [13] which should be as strong as the substrate material being joined [11]. The process can be distinguished by four key steps: i) the incubation period (when the electrical current is introduced and the joint has no strength), ii) the joint formation and consolidation (after fusion, the gap is filled and the PE is molten), iii) the plateau region (the strength stabilizes

with respect to fusion time), and iv) the cooling period (crystallization). A fifth step, the degradation time, can appear if the heating is not stopped soon enough, indeed after a precise time, the joint loses strength as the heating continues [11]. This defect is called over welding and is due to too much energy or too high a temperature during the EF process.
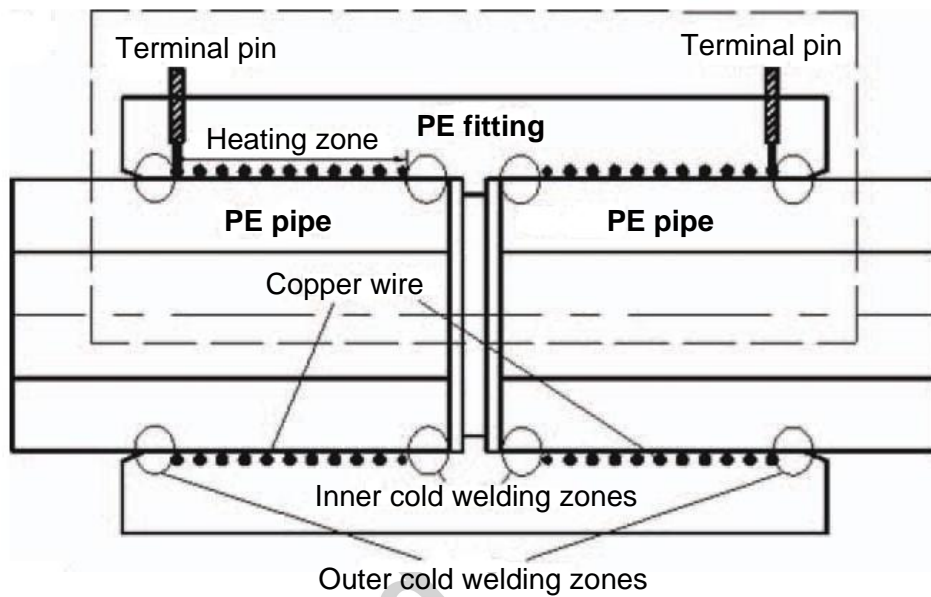
Fig 1 – Schematic sketch of an EF joint [8].

Three other kinds of defect that can affect EF joint quality are: i) structural deformity (misalignment, inadequate insertion, wire dislocation), ii) voids (discontinuity of the joint) and iii) poor fusion interface (due to contamination of the fusion interface by dust or debris or to lack of energy or time during welding (cold welding)). Fig 2 shows three failure modes that can occur as a result of defects in EF joints, the failure mode is known to depend on the length of the cold welding zone and the input energy, but it remains largely inexplicable in practical applications [8].
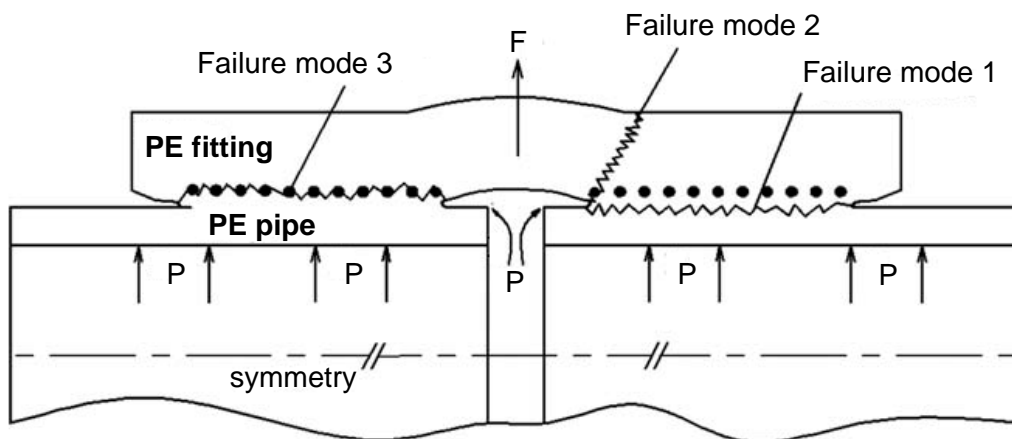
Failure mode 1: cracking through the fusion interface
Failure mode 2: cracking through the fitting
Failure mode 3: cracking through the copper wire interface

Fig 2. Failure modes of an EF joint [8].

The most recognised alternative to EF welding is butt fusion (BF) welding, which involves heating the joint areas with a hotplate and then contacting the interfaces to initiate intermolecular fusion for bonding [14]. BF has some key advantages over EF such as higher yield strength and ultimate tensile strength and greater elongation to failure [15]. Other fusion techniques resulting from external heat sources can also be used to join PE pipes [14], the source can be a laser (Visible Through Transmission Laser Welding (TTVLW) [16]), or a stream of hot air [13] for example. Generally, these welding methods are fast and quite simple to use, but their capital cost is a big drawback.

Mechanically locking the pipes to one another using flanged joints is also a feasible solution [14] and enables full joint strength to be obtained immediately as well as a joint which is easily disassembled. However, these types of joints do not have good sealing capability and are very likely to cause stress concentrations.

The high cost of BF welding and the huge inconveniences of mechanical techniques have certainly enhanced the infatuation for EF welding. Although EF is now the most widely used jointing technique for PE pipe, as discussed above, it also has its own drawbacks. According to the PPDC's (Plastic Pipe Database Committee) latest 2015 status report, 65% of PE pipeline failures or leaks were due to problems with EF joints (11.4%) and fittings (53.6%), with the remaining 35% being attributed to faults in the pipeline itself (31.8%) or not being recorded (3.2%) [17].

One potential alternative to EF welding that is receiving increasing attention is adhesive bonding [18-19]. Traditionally, adhesives have often been overlooked as a potential jointing method due to the difficult nature of bonding PE. PE belongs to the polyolefin group of plastics which are notoriously difficult to bond due to their low surface free energy, which prevents adhesives from successfully wetting their surface. For example the surface free energy of PE is only 31 mJ/m^2 [20] which is lower than that of typical epoxy based adhesives which have values in the range 45 to 50 mJ/m^2 [21].

There are several pre-treatment methods that can be carried out on PE in order to increase its surface free energy and thus improve adhesive joint strength. Chemical treatment is possibly the most effective of these techniques; the application of sulfuric (or chromic) acid is known to remove the weak boundary layer and introduce polar groups to improve adhesive strength [22-23] but is slower and more hazardous than traditional mechanical techniques [21]. Another efficient treatment is exposure to UV light in the presence of a solvent [6, 22], which can increase the usable

surface and its oxygen content, increasing joint strength by up to a third, but it is quite expensive and time consuming to perform [24]. Flame treatment is another technique that has been shown to work well on polyolefins such as polypropylene (PP) and PE [22, 25], this method is preferable to mechanical treatments but can be costly and it is more suitable for use on large parts [18]. Various mechanical treatments also exist, from manual abrasion to sandblasting or gritblasting [21-22], and can significantly enhance adhesion. Plasma treatment methods such as plasma surface pretreating [26-28], Atmospheric Pressure Plasma Jet (APPJ) [29] and glow discharge plasma [5, 30] are also very popular. Within this group, low temperature plasma and corona discharge have been reported to be the two best methods to improve PE adhesion [18] but their high cost and relatively low shelf life can make them less attractive.

In addition to treatment of the PE substrate surface, recent improvements in adhesive technology have also meant that the ability of the adhesives themselves to bond to low energy or contaminated surfaces has also improved. For example heat cured epoxies now have good solubility of oil contaminated surfaces when compared to water based adhesives, which simply form a hardened film that slips on top of the oily surface. Scavengers can also be added to most epoxy adhesives to help further disperse surface contamination.

With these recent advances in adhesive science, coupled with the increasing range of effective surface pre-treatments, the production of adequate structural adhesive bonds between MDPE pipelines may now be feasible as an alternative joining method to traditional EF welding. Previous work by the authors has highlighted the

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potential of using acrylic adhesives to successfully bond MDPE pipeline and tapping tees [19]. This work focussed on testing the shear strength and impact resistance of the adhesively bonded MDPE joints at room temperature and therefore did not account for variations in soil temperature which occur throughout the UK's seasonal climate. This current study will therefore focus specifically on the effect of low temperature testing, down to -10°C, on the shear strength of adhesively bonded MDPE pipe. In particular, the influence of curing time, curing temperature, clamping load and test temperature, on the shear strength of the bond are investigated.

2. Material and Methods

The tensile lap-shear specimens were prepared by bonding two MDPE (PE80-yellow) substrates with a two-part methylmethacrylate (MMA) based structural adhesive (Weicon Easy-Mix PE-PP 45), which was applied using the manufacturers dispenser pistol and mixing nozzle. MMA based adhesives have a crosslink density between that of epoxy and polyurethene adhesives which makes them particularly well suited for creating fast, high strength bonds on low energy plastics like PE and PP, whilst also producing a joint with good flexibility. The key charaterisitcs of the substrate and adhesive materials are given in Table 1.

Table 1 – Key properties of the MDPE substrate and acrylic adhesive.

MDPE substrate (PE80 yellow gas pipeline)	
Type	PE80 yellow gas pipeline
Brand	GPS PE Pipe Systems
Dimensions	180 mm diameter, 18 mm thick
Density	0.93-0.95 g/cm^3
Tensile strength	14-22.8 MPa
Acrylic adhesive	
Type	Two-component construction adhesive based on methylmethacrylate (MMA)
Brand	WEICON Easy-Mix PE-PP 45
Mixing ratio by volume	10:1 (resin/hardener)
Density	1.07 g/cm^3
Viscosity at +20°C	45 mPa.s
Pot life (10 ml of material at +20°C)	2-3 min
Glass transistion temperature (Tg)	35 °C
Processing temperature (optimal)	+20 to +25°C
Curing Temperature	+15 to +70°C
Curing time at 20°C (for PP substrates)	2-3 hrs – handling strength (35% of final)
	6 hrs – mechanical strength (50% of final)
	24 hrs – final strength (100% cured)
Coulour before curing	Colourless, translucent
Colour after curing	Yellowish, transparent
Average maximum expansion	5.3%
Average Shore hardness D	55
Average tensile strength	13 MPa
Average lap shear strength	2.8 Mpa – when bonding low density PE
	7.4 Mpa – when bonding high density PE
Average peel strength	2.9 MPa
Temperature resistance	-50 to +80°C
Coefficient of thermal expansion	125 x 10^{-6}/K – below Tg
	170 x 10^{-6}/K – above Tg

The MDPE substrates were cut from the pipeline using a bandsaw into strips of dimensions 160 x 25 x 18 mm thick, which were then assembled and bonded in a lap shear configuration using the MMA adhesive, with a 50 x 25 mm bond area in accordance with ASTM D1002-99 [31], see Fig 3. Prior to assembly, the bond area surfaces of the substrates were cleaned using Weicon solvent spray surface cleaner and wiped dry with a clean cloth. The adhesive bond line thickness was indirectly controlled to a maximum of 0.5 mm via variation in clamping pressure (as discussed in the following section) and to a minimum of 0.2 mm via glass particles within the adhesive.
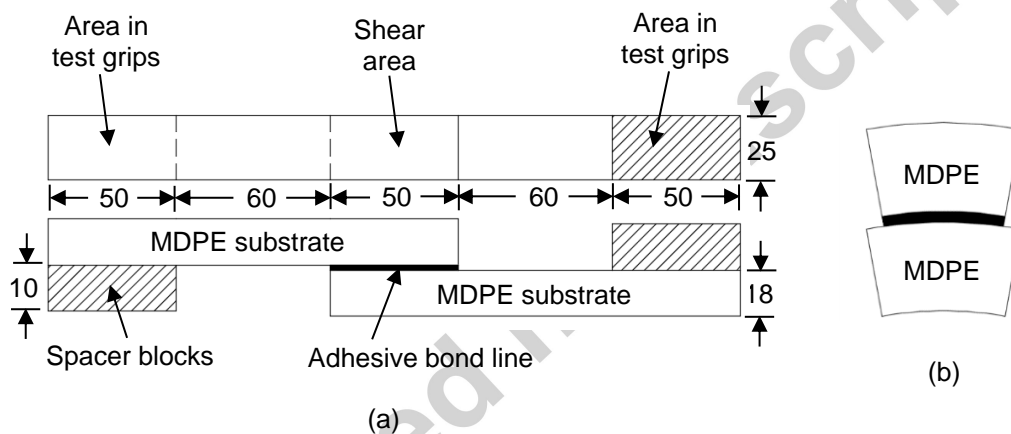
Fig 3 – (a) Specimen geometry for lap-shear adhesive joint (dimensions in mm), (b) Cross sectional view showing curvature of substrates.

A total of 75 tensile lap-shear specimens were prepared in this manner as detailed in Table 2. To determined the effect of clamping pressure on the shear strength of the adhesive bond, the first group of 25 samples (batches 1A-E) were all cured at room temperature (20°C) for 16 hours under various loads from 0 to 2 kg, as applied by free weights positioned on the bond area. Once the optimum clamping pressure was determined, the next group of 25 samples (batches 2A-E) were cured under this pressure at 20°C for various durations between 4 to 48 hours in order to study the

effect of curing time. Finally, once the optimum curing time and clamping pressure were established, the remaining group of 25 samples (batches 3A-E) were prepared under these conditions at various curing temperatures from -10 to +20°C in order to study the effect of curing temperature on the shear strength of the adhesive. All group 3 batches, except those cured at room tempeture (batch 3E), were cured in a LEC R450CW refridgerator and their temperature was continuously monitored using a Digitron type 2029T thermometer and K-Type thermocouple positioned on the edge of the bond area during both the curing and testing phases.

Table 2 – Lap-shear specimen preparation conditions.

Specimen ID		Sample size	Curing conditons		
Group	Batch		Temp (°C)	Time (h)	Clamping Pressure (Pa)
	A	5			0
	B	5			340
1	C	5	20	16	740
	D	5			1180
	E	5			1570
	A	5		4	
	B	5		8	
2	C	5	20	16	1180
	D	5		24	
	E	5		48	
	A	5	-10		
	B	5	-5		
3	C	5	0	24	1180
	D	5	5		
	E	5	20		

All tensile tests were carried out at 20°C in accordance with ASTM 1002-99 [31] using an Instron 3382 tensile testing machine with a 100 kN load cell under a crosshead speed of 2.0 mm/min. To keep temperature rise to a minimum during testing, all specimens cured at low temperatures (batches 3A-D) were insulated using a custom made enclosure, manufactured from DOW STYROFOAM insulating foam, see Fig 4. Following testing the lap-shear strength of each specimen, in Pascals (Pa), was calculated as the measured peak load divided by the true surface area of the bond, as measured prior to testing. Statistical analysis of the results was performed using a one way ANOVA test incorporating Games-Howell pairwise comparisons using Minitab 17 software. All samples were closely observed during testing and the fractured surfaces of the joints were examined afterwards using a Nikon LV-100 upright microscopy system.
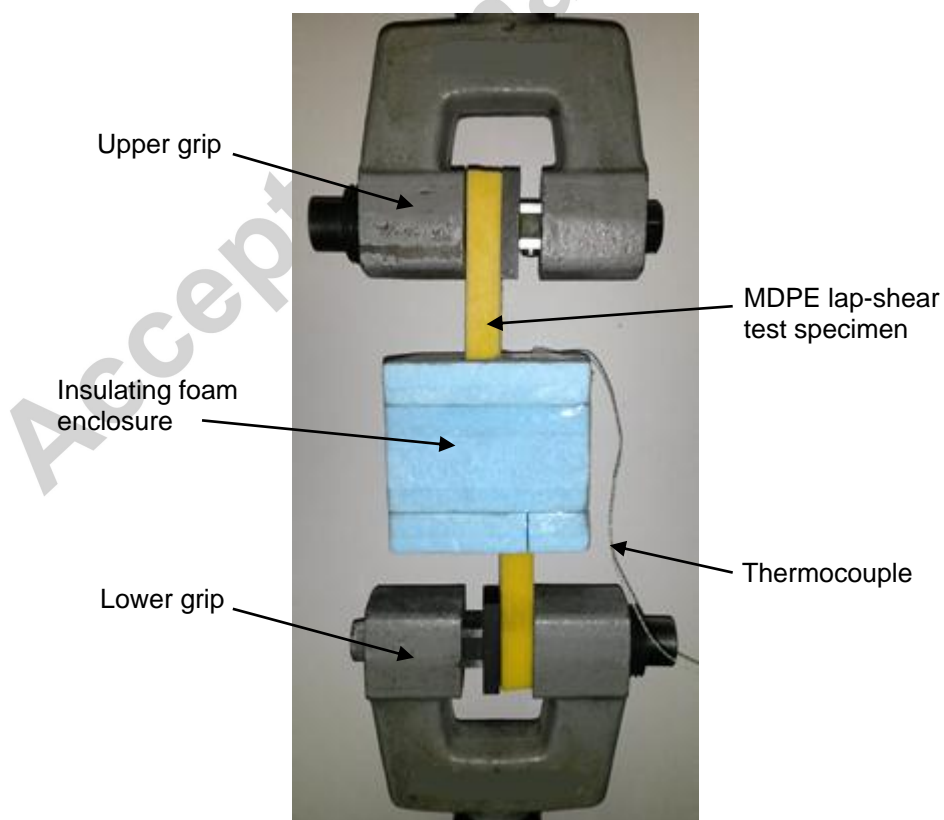

Fig 4 – Experimental setup for lap-shear tensile tests.

3. Results and Discussion

3.1 Effect of varying clamping pressure

Typical lap shear stress-strain curves for adhesive joints bonded under various clamping pressures of 0 to 1570 Pa (batches 1A-E) for 16 hours at 20°C are shown in Fig 5.
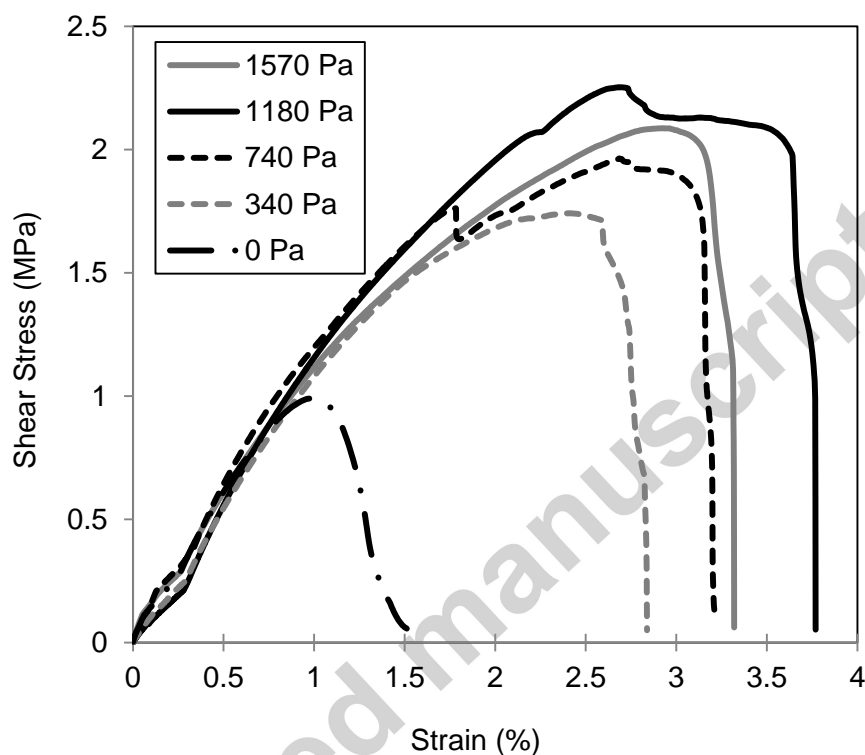

Fig 5 – Typical lap shear stress-strain curves for the adhesive joints bonded under various clamping pressures of 0 to 1570 Pa.

The majority of samples exibited similar behaviour with a region of strain in the MDPE substrates of 2 to 4% before the adhesive failed suddenly in a brittle nature at stresses of 1.5 to 2.8 MPa, depending on the applied clamping force. One key exception to this trend was the failure mechanism of the samples bonded with no clamping force (batch 1A) which were very inconsistent. Of the five samples in this batch, two failed to form a bond with sufficent strength to be tested and the remaining three failed at relatively low stresses of 1 to 1.7 MPa. In additon to this

behavoir, there were also several samples which exibited two peaks in stress at around 1.5 to 2% and 2.5 to 3% strain, such as the sample clamped under 740 Pa of pressure reported in Fig 5. This double peak was found to be caused by an intial splitting of the bond line followed by catostrophic failure of the adhesive joint area.

Results of mean and scatter of lap shear strength for the batches of 5 samples bonded under various clamping pressures (batches 1A-E) are reported in Fig 6.
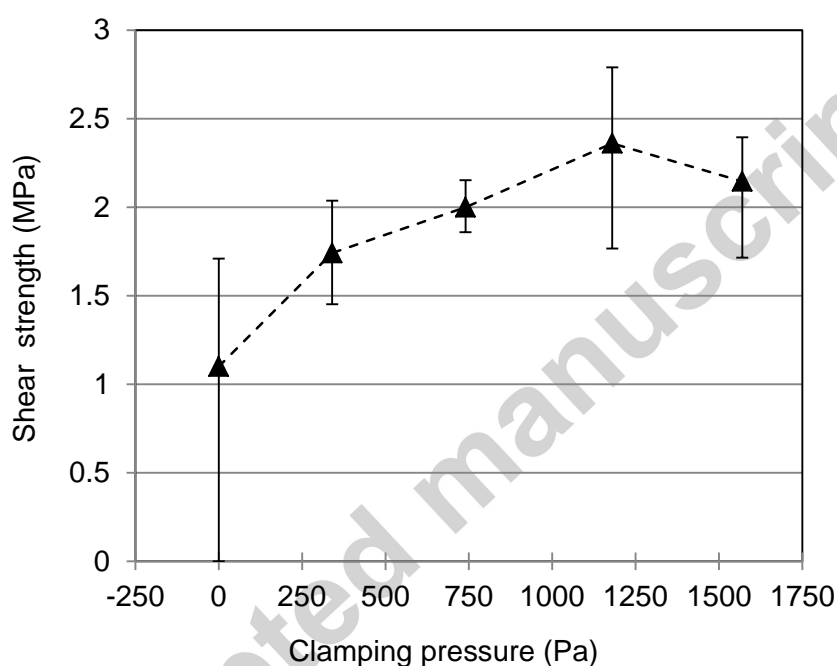
Fig 6 – Lap shear strength of the adhesive joints bonded under various clamping pressures of 0 to 1570 Pa.

There appears to be a clear increase in the average shear strength with increasing clamping pressure from 1.1 MPa at zero pressure to 2.36 MPa at 1180 Pa of pressure, after which the average strength reduces to 2.14 MPa at the maximum clamping pressure of 1570 Pa. Statistical analysis of the results (excluding results at zero clamping pressure) showed that there was a significant difference, at the 95% confidence level, between the results at 340 and 1180 Pa, see Table 3. There was

no significant difference between the results at 740, 1180 and 1570 Pa or between the results at 340, 740 and 1570 Pa.

Table 3 – One-way ANOVA and Games-Howell pairwise comparison results for the effects of clamping pressure and curing time on the lap shear strength of the adhesively bonded joints.

One-way ANOVA

Factor	Levels	Values	DF	F Value	P Value	R-sq (%)
Pressure (Pa)	4	340 Pa 740 Pa 1180 Pa 1570 Pa	3	3.84	0.046	43.98
Time (h)	5	4 h 8 h 16 h 24 h 48 h	4	12.06	0.02	80.52

Games-Howell Pairwise Comparisons (95% confidence)

Factor	Level	N	Mean (MPa)	Grouping*		
Pressure (Pa)	1180 Pa	5	2.361	A		
	1570 Pa	5	2.146	A	B	
	740 Pa	5	2.0002	A	B	
	340 Pa	5	1.741		B	
Time (h)	24 h	5	2.720	A		
	16 h	5	2.296	A	B	
	48 h	5	2.1709		B	
	8 h	5	1.938		B	C
	4 h	5	1.117			C

*Means that do not share a letter are significantly different

As previously discussed, only 3 out of the 5 samples bonded under zero clamping pressure had sufficient strength to be tested, suggesting that a minimum force must be applied to the bond area to establish sufficient adhesive contact. Once this is established further increases in pressure up to 1180 Pa serve to improve this

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adhesive contact and strengthen the bond and at pressures beyond this, the adhesive starts to be expelled from the joint reducing the bond line thickness and strength. Based on this result all further samples were prepared under a clamping pressure of 1180 Pa.

3.2 Effect of varying curing time

Results of mean and scatter of lap shear strength for the batches of 5 samples bonded under various curing times from 4 to 48 hours (batches 2A-E) at 20°C under a clamping pressure of 1180 Pa are reported in Fig 7.
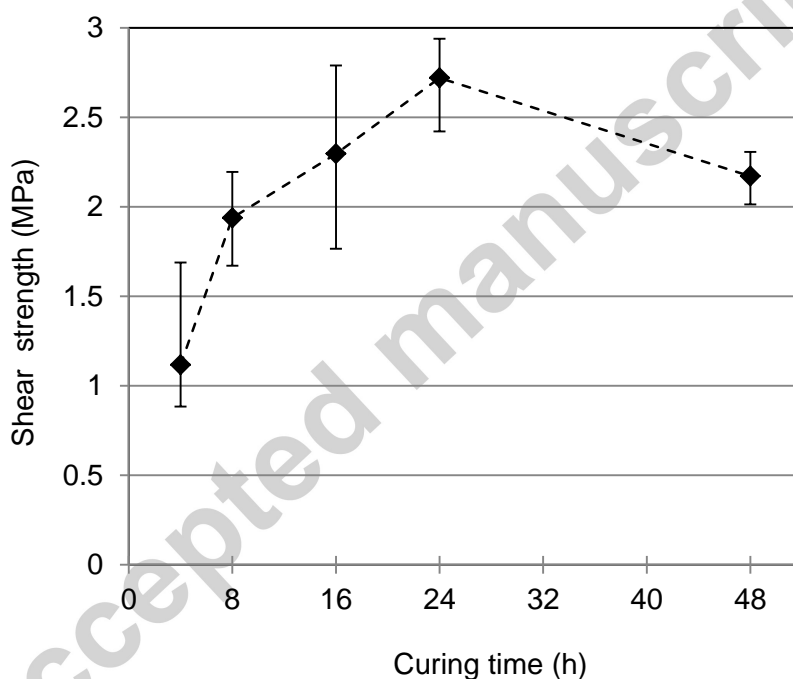

Fig 7 – Lap shear strength of the adhesive joints bonded for various curing times of 4 to 48 hours.

After only 4 hours of curing the bonds have gained sufficient strength to be tested but their shear strength results are relatively low in the range 0.9 to 1.7 MPa. This strength increases with curing time to a maximum range of 2.4 to 2.94 MPa after 24 hours before reducing to 2 to 2.3 MPa after 48 hrs. Statistical analysis of the results

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showed that there was a significant difference, at the 95% confidence level, between the results at 24 hours and those at 4, 8 and 48 hours, see Table 3. There was no significant difference between the results at 24 and 16 hours or between the results at 8, 16 and 48 hours or between the results at 4 and 8 hours.

This variation in bond strength with increasing curing time has been reported in several previous studies [32-35], with some results suggesting that a maximum strength is achieved at a given time and remains around this value with further increase in time [33], whilst others have reported a slight decrease in strength after reaching this maximum value of strength and have related this to phenomena such as adhesive oxidation reaction [34] or over drying [35]. In this work, although the bond strength begins to decrease slightly after 24 hours of curing, further experiments would be required to confirm that this does not continue beyond 48 hours. Based on this result all further samples were prepared under a clamping pressure of 1180 Pa and cured for 24 hours.

3.3 Effect of varying curing temperature

Typical results of temperature rise for samples cured at temperatures of -10 to +5°C (batches 3A-D) and enclosed in insulating foam, following removal from low temperature storage, are shown in Fig 8. The temperature rise is quite linear across the 300 second time period in the range 0.001 to 0.0015°C/sec with decrease in curing temperature from +5 to -10°C. The temperature of the specimens at the start (t=60 sec) and end (t=180 sec) can also be seen in Fig 8. The average temperature during testing is used as the test temperature in the following section.
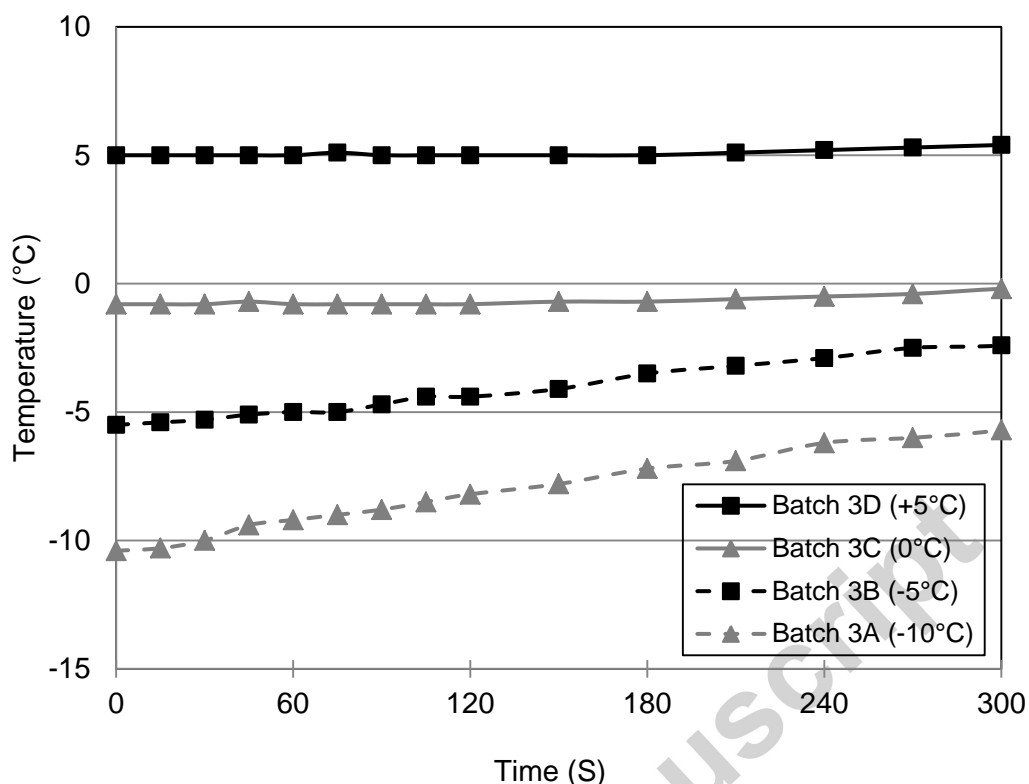
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Fig 8 – Temperature rise of samples following removal from storage at various curing temperatures of -10 to +5°C

Figure 9 shows lap shear stress-strain curves for test samples from batches 3C, D and E bonded under a clamping pressure of 1180 Pa and cured for 24 hours. No results are reported for batch 3B, which was cured at -5°C, as all five samples in this batch had insufficient strength and broke during handling prior to testing. A similar result was achieved for batch 3A, which was cured at -10°, with only one out of the five samples having sufficient strength to be tested, which was very low, failing at a stress of 0.105 MPa at 0.18% strain.
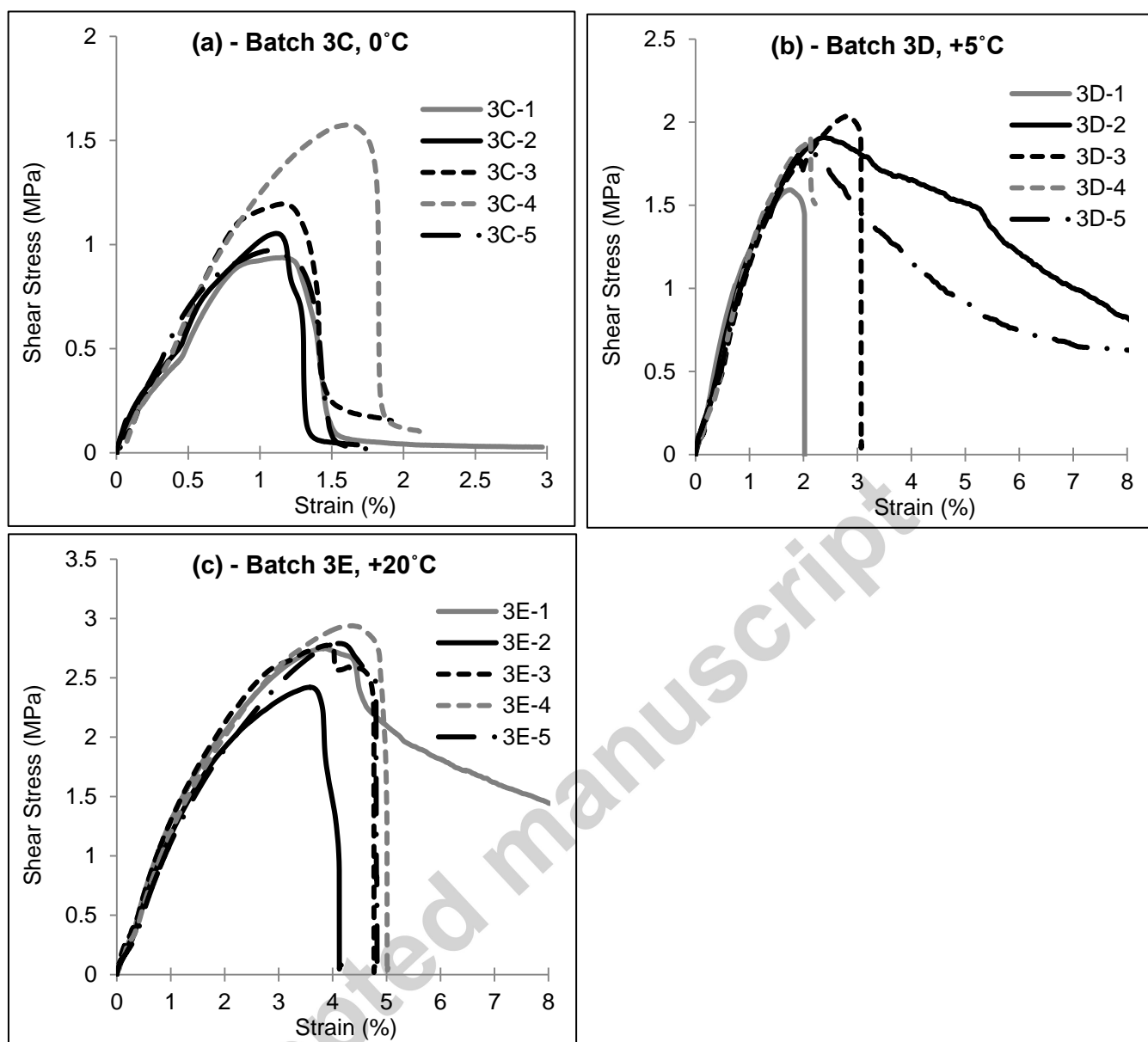
Fig 9 – Lap shear stress-strain curves for the adhesive joints cured under temperatures of 0, +5 and +20°C

The results for the samples cured at higher temperatures of 0 (Fig 9a), +5 (Fig 9b) and +20°C (Fig 9c) were much improved, with all 5 samples in each batch being successfully tested. The majority of test samples failed in a similar nature with a region of strain in the MDPE substrates before the adhesive failed suddenly in a brittle nature, with both the level of stress and strain at failure increasing with

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increase in curing temperature. There were some exceptions to this type of failure, sample numbers 3D-2 and 3D-5 (Fig 9b) and 3E-1 (Fig 9c), in which, after reaching peak stress, the MDPE substrates continued to strain to 8% (after which point the test was stopped) and the the adhesive joints did not fail.

A summary of the results of mean and scatter of lap shear strength for the batches of 5 samples bonded under various curing temperatures from -10 to +20°C (batches 3A-E) are reported in Fig 10.
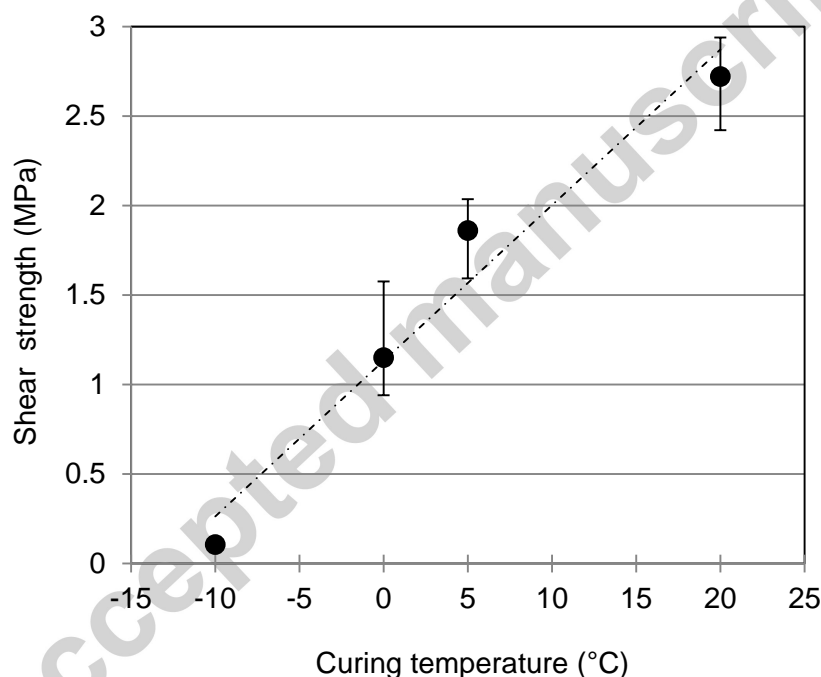

Fig 10 – Lap shear strength of the adhesive joints cured under various temperatures of -10 to +20°C

There is a steady decrease in shear strength with decrease in curing temperature from a mean value of 2.72 MPa at +20°C to 1.15 MPa at 0°C. Below zero degrees the strength of the bonded MDPE substrates is significantly reduced; no samples

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bonded at -5°C had sufficient strength to test and only one sample bonded -10°C was tested, which had very low strength of 0.105 MPa. Although EF welding is known to produce joints as strong as the parent materials being joined [11], (in this case the MDPE pipeline has a strength of 14 to 22.8 MPa, see Table 1), previous work by the authors has shown that adhesive lap shear joint strengths as low as 1.75 MPa can be sufficient to successfully joint MDPE pipeline [19]. Therefore the results in Fig 10 for samples cured at +5 and +20°C are within this range but those cured at lower temperatures of 0, -5 and -10°C are not.

Previous work regarding low temperature testing of adhesive joints has shown that it is possible to maintain joint strength at sub-zero temperatures. In fact, with correct control of bondline thickness, joint strength has been shown to increase down to test temperatures of -40˚C, as the adhesive becomes stiffer [36]. However, with further decreases down to cryogenic temperatures (-252˚C), adhesives can become too stiff, leading to brittleness and significant reductions in bond strength [37]. In this current work it is believed that the reduction in bond strength is a function of the decrease in curing temperature rather than the decrease in test temperature. This theory is supported by the results in Fig 11, which shows the typical failure modes of the bonds cured at various curing temperatures from -10 to +20°C (batches 3A-E), following post test inspection of the fractured surfaces. Typical images of the fractured surfaces are shown Fig 12.
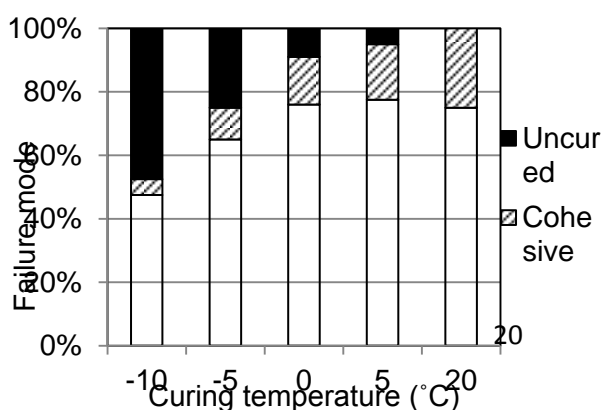
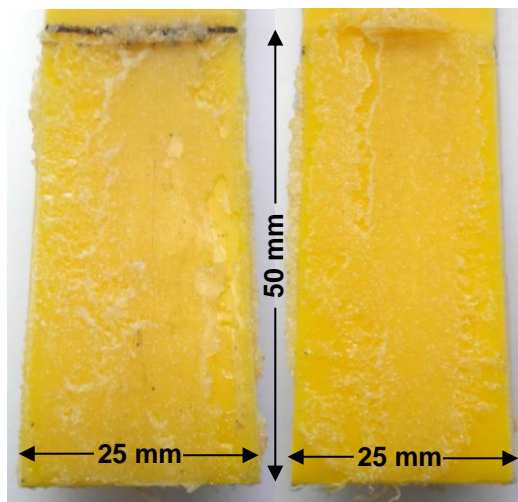Fig 11 – Typical failure modes of the adhesive joints cured under various temperatures of -10 to +20°C

Fig 12 – Typical images of the fractured lap joint surfaces.

The analysis of the joints showed that at low curing temperatures a considerable proportion of the adhesive remained uncured after 24 hours. On average this accounted for 48% of the bond area for the samples cured at -10°C and steadily reduced to 5% for the samples cured at +5°C, and 0% for the samples cured at +20°C. This result is in line with that of previous work using acrylic resin, which reports a considerable increase in the percentage of uncured adhesive with decrease in curing temperature from +25°C to +1°C [33]. Conversely the proportion of adhesive and cohesive failure reported in Fig 11, both increased with increasing curing temperature and the ratio of cohesive to adhesive failure increased from 1:10 at -10°C to 1:3 at +20°C. Although this ratio of apparent adhesive bond to cohesive strength is still quite low, it is typical when trying to bond PE to PE due to its inherent low surface energy [19-20].

4. Conclusions

This work investigated the low temperature lap-shear strength performance of adhesively bonded MDPE pipe as a potential replacement for conventional electrofusion welded joints. Samples of MDPE were first bonded under a series of clamping pressures for a range of curing times at room temperature before their tensile lap-shear strength was tested. The optimum conditions at room temperature for pressure and time were determined to be 1180 Pa and 24 hours respectively and samples prepared under these conditions achieved shear strength values in the range 2.4 to 2.94 MPa.

Following this, further samples were then prepared under these conditions at a range of temperatures of -10, -5, 0, +5 and +20°C. It was found that a decrease in curing/testing temperature to zero degrees resulted in a steady decrease in the lap-shear strength performance of the bonded joints from a mean value of 2.72 MPa at +20°C to 1.15 MPa at 0°C. Below zero degrees the strength of the bonded substrates was significantly reduced; no samples bonded at -5°C had sufficient strength to test and only one sample bonded -10°C was tested, which had very low strength of 0.105 MPa.

Although some promising lap-shear strength results have been achieved for MDPE samples bonded and tested at room temperature, which are in line with previous work [19], the reduction in strength with curing and test temperature poses a significant barrier to the development of adhesive bonding as a realistic replacement for electrofusion welded polyethylene gas pipelines. To try and tackle this problem, future work will focus on reducing the percentage of uncured adhesive present at low

curing temperatures by increasing or accelerating the curing cycle time before the joint is exposed to sudden reductions in temperature, as well as considering modification of the adhesive composition to improve its low temperature performance.

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