Deformation and failure in polyethylene: correlation between mechanisms of creep and fatigue

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Fatigue and creep tests have been conducted on medium density gas- and water-grade pipe materials using a longitudinally notched pipe geometry with internal pressurization. The failure in both types of test was brittle compared to the failure occurring when the unnotched pipe is exposed to a constant intense pressure. Further to this superficial similarity, fractographic studies revealed creep and fatigue failures to have many features in common. Thus it is perhaps not surprising that fatigue and creep lifetimes have been found to correlate well. The findings of this study give support to the concept of adopting fatigue as a quality control method in the production of plastic pipe.

1. Introduction

The use of polyethylene pipe in both gas and water distribution systems is increasing dramatically. However, in order to manufacture pipe of a suitable quality it is necessary to understand the influencing factors which determine pipe longevity and to use this knowledge to implement and enhance design and manufacturing standards. It follows that a method of quality control is needed which ensures the production of pipe to the required standard. Traditionally this has been done via a test based upon static internal pressurization whereby design is based upon creep regression curves. The virtual absence of field failures since the introduction of pipes into service has meant that there is a resistance to any change from the tried system. However, owing to the massive improvement in pipe resins the test is no longer appropriate and a highly accelerated lifetime test is required for the introduction of on-line quality control testing. Fatigue offers such an opportunity especially as there is evidence for good correlation between creep and fatigue data [1].

In the absence of a thorough scientific understanding of the causes of lifetime termination, it is necessary to select a quality control test which in the first instance yields similar types of failure to those seen in service. Thus as the most damaging and premature failures involve brittle-type fracture a short-term test is required which produces brittle-type failure of the pipe. It is also of value to have a test method which is representative of in-service conditions. Thus we are currently engaged in the research and development of a fatigue testing method which is suitable for producing brittle-type failures on a timescale of hours rather than the weeks more usually associated with creep tests.

In this method, sections of pipe are exposed to cyclic internal pressurization by water. The pipes are

notched to inhibit failure by the more prevalent mode of ductile flow under high or monotonically increasing pressure. Both creep and fatigue tests were conducted on the same machinery.

Critical to the implementation and usefulness of fatigue testing as both a quality control method and as an assessor of lifetime expectancy is the exact nature of failure in fatigue and comparison between fatigue, creep and in-service failures. There is a dearth of actual long-life service failures and those that do occur are usually due to an abnormal inclusion in the material or poor installation. However, their presence points to the need of having a basic material resistant to brittle-type failure. Superficially, in-service failures have many of the features seen in cracks grown monotonically, but the service load is unlikely to be that constant and examined at this level there are examples of fatigue failures of similar appearance [2]. It is of scientific and commercial interest, therefore, to examine these apparent similarities in more detail. This paper discusses the early findings of a wide investigation into the nature of creep and fatigue failures in polyethylene pipe and pipe materials and provides a useful insight into failure mechanisms which are not as simple or straightforward as those previously suggested in the literature.

2. Materials and methods

2.1. Materials

The sections of pipe tested were principally manufactured from medium-density polyethylene. Both yellow gas- and blue water-pipe grade material were used. The materials were commercial-grade Rigidex manufactured by B.P. and the chemical and physical structure conforms to that standard. Pipes made from other materials were used for comparison.

2.2. Testpieces

Testpieces for fatigue and creep tests were cut from sections of commercial-grade pipe of nominal outside diameter 90 mm and ratio of pipe diameter to pipe wall thickness (SDR) of 11. A longitudinal notch was cut in the outside of the pipe. Methods of notching and size of the testpieces have been reported elsewhere [3].

2.3. Testing

Fatigue and creep tests were conducted over a range of internal pressures. A square-wave load form was used at 0.4 Hz. The rise time was 35 ms (time from minimum to maximum pressure). Justification for using these conditions and detailed reports of test results over a wide range of test conditions are published elsewhere [4]. Fatigue was conducted in water at $80 °C$.

2.4. Fractography

Samples were prepared for electron microscopy by sputtering with gold/palladium before viewing in a Jeol 200 scanning electron microscope.

Transverse sections of the fracture zone were taken by immersing large sections of the pre-notched area of the pipe in liquid nitrogen for i0 min prior to producing transverse fractures by placing a sharp bladed instrument on the section required and impacting with a mallet. The resultant fracture surface prepared as above was then viewed in the SEM.

The resultant micrographs are shown in Figs 1-14.

3. Results and discussion

Fatigue fracture in many materials capable of plastic flow is accompanied by a stepwise progression of crack advance which leaves a historic record of the applied stress intensity in the form of striations on the fracture surface. These are caused by localized ductility at the crack tip and demark positions of crack front advance and arrest with each load cycle.

Striations have also been reported on the creep and fatigue fracture surfaces of polyethylene. Two types of striation have been widely reported: a "macrostriation", which can be seen with the naked eye, occurs widely in both creep and fatigue whilst microstriations, which can only be observed microscopically, have been more widely documented in fatigue than in creep. Macrostriations have been associated with the growth and breakdown of a craze at the crack tip in both creep and fatigue modes of fracture. Another interesting feature which fatigue and creep fracture modes share is the propensity of the crack front to branch above and below the fracture plane. This type of deformation has previously been ascribed to a localized shear deformation zone in the case of creep [5]. However, Fig. 1 shows branching fracture/deformation zones above and below the fracture plane of a fatigue sample. Fig. 1 is a liquid nitrogen fracture surface on a transverse plane to the fatigue crack. As well as branching "cracks", a crazelike structure is seen at the crack tip. Fig. 2 shows a

Figure 1 The tip of a fatigue fracture showing a craze-like form (CLF) and branching fracture features (BFF).

Figure 2 Higher magnification view of the CLF in Fig. 1.

Figure 3 Craze fibrils bent over in the CLF of a pigmented carbonblack material.

higher magnification view of the craze-like zone. Fig. 3 shows a similar zone in a carbon-black pigmented material. It appears in Fig. 3 that the craze fibrils are bent over. Although this is at first surprising, given that the test piece was always under an applied tensile load, it becomes less so if one considers the high local extensions that must occur at the crack or craze tip. These micrographs give support to the mechanisms of fatigue crack growth in polyethylene as proposed by Zhou and Brown [6]. However, the method adopted here, of taking transverse sections by fracture in liquid nitrogen (TFLN), provides a useful way of relating the sub-surface structure of fracture surfaces to topographical features on the fracture surface itself without the inherent difficulties of etching

techniques which tend preferentially to etch away edges. Etching may also remove "amorphous" fractions and stressed material possibly giving rise to an apparent voided structure when this is not present. Thus Fig. 4 shows a TFLN of a region of a fatigue fracture surface on which microstriations are the prevalent topographical feature. It is evident that the apparently solid coherent fracture surface is undermined by a highly deformed zone of material which is similar in appearance to the bottom half of the crazelike structure shown in Figs 2 and 3. This is in contradiction to the accepted mechanism proposed by White *et al.* for the formation of microstriations in the fatigue of polyethylene $[7, 8]$ which involves fatigue crack propagation by limited plastic flow and rearrangement of lamellae at the crack front (a mechanism more in keeping with the classical fatigue mode of metals). Indeed it is easy to understand how the coherent fatigue fracture surface can be misleading as the surface alone gives no easily interpreted clue as to underlying structure. However, armed with the evidence of Figs 2–4 one may assume an underlying structure to fatigue fracture surfaces (FFS). For example, Fig. 5 shows an apparently fiat FFS of a water-pipe grade material; if a piece of material is cut from an adjacent area of the fracture surface and then bent uniformly at right angles to the direction of crack propagation, the surface transforms to that appearing in Fig. 6. Again, macrostriations appear, showing that a mechanistic

commonality exists between failure surfaces of superficially different appearance. It is also of interest to note that branching fracture features (BFF) are not evident in this particular case where the FFS appears smooth, implying a competitive relationship between the mechanisms of formation of BFF and FFS. Thus although others have dismissed BFF as artefacts in the case of creep [5] we believe them to be relevant to the failure in both creep and fatigue. (It may be noted that BFF have been associated with a crack blunting mechanism in the plane-strain fracture of polyethylene [9, 10] and have been reported in the fatigue of both polyethylene [11] and glassy thermoplastics [12].)

Applying the technique of TFLN to creep fracture surfaces reveals that there is also underlying structure to apparently brittle fracture surfaces. Fig. 7 shows the initiation zone of a creep fracture initiating at a notch root (the fracture having initiated at two different sites at the notch root). The initiation zone appears very smooth, however, TFLN reveals that the sub-structure of this surface has the craze-like form (CLF) shown in Fig. 8. Failure has thus occurred at the craze/solid interface. This zone of failure gives way to a rougher zone further away from the notch root which has no uniform sub-surface CLF, thus failure is probably propagating through the CLF, perhaps jumping from interface to interface, the transition zone between smooth and rough fracture being shown in Fig. 9. Further on, the FFS takes on the relatively

Figure4 Section through a fatigue fracture surface upon which microstriations are the prevalent surface features.

Figure 6 Banding appears after bending open the flat failure surface shown in Fig. 5.

Figure 5 An apparently flat fatigue fracture surface. Notch root initiation site is at bottom right, and a termination impact fracture at top left.

Figure 7 The origin of creep fracture (notch, top right).

Figure 8 Close-up view of a section of the origin of the creep fracture surface shown in Fig. 7 showing the underlying CLF.

Figure 10 Further away from the notch root, the creep fracture looks relatively smooth, with markings similar to those seen on the fracture surfaces of PMMA.

Figure 9 Final fracture in this creep specimen occurs by separation at the craze-solid interface.

smooth appearance seen in Fig. 10. This is not dissimilar in nature to "parabolic" fracture in PMMA [8]. TFLN (Fig. 11) shows this zone to be undermined by a very similar CLF to that seen beneath the zones of microstriations on FFS. Again this corresponds to failure down the CLF mid-plane. It thus appears that both creep and fatigue fracture surfaces of materials in our investigations maybe interpreted in terms of the growth and breakdown of a CLF. The situation in fatigue is complicated by the unloading cycle, which depending on loading conditions (such as the degree of offset from the mean pressure) may accentuate CLF breakdown by buckling mechanisms as outlined by Zhou and Brown [6].

Thus there appears strong evidence to suggest that the underlying mechanisms of creep and fatigue fracture in polyethylene have much in common. This is consistent with the good correlation found between creep and fatigue lifetimes [1]. We have also encountered strong evidence for an interaction between filler particles and the mechanism of brittle fracture in the same materials as those in the above figures. Fig. 12 shows an initiation zone of brittle fracture. Islands of material are ringed with white borders. When this surface is tilted on its side the islands are shown as raised plateaux. Higher magnification micrographs show holes corresponding to the known size of filler particles in the middle of the plateaux (Fig. 13). It is, therefore, quite plausible that filler particles have initiated fracture on different planes in the initiation

Figure 11 TFLN shows the smooth area seen in Fig. 10 to be undermined by a fibrillar structure suggesting that the failure in this region occurred by separating along the CLF mid-plane.

Figure 12 "Mackerel" markings are islands of material surrounded by drawn material and demark the initiation of fast fracture.

zone when the crack is accelerating and has not reached a velocity such that the propagating stress intensity can escape the "drag" of the local stress fields associated with filler particles, Further away from the initiation zone the crack becomes flat and planar. Thus although we have no direct evidence at present of interaction between filler particles and creep or fatigue fracture this remains an important part of the investigation. The practical importance of interpreting fracture surfaces clearly shows the need to investigate the nature of the deformation and fracture mode

Figure 13 Higher magnification view of "mackerel" markings.

transitions which appear to influence the diversity of failure characteristics observed in both creep and fatigue. In addition to stress-activated transitions, isothermal/adiabatic transitions maybe important. For example, if shear bands do form in continuous creep tests as reported elsewhere [5] then this conflicts with theoretical considerations which suggest that in the absence of a contiguous microstructure, in which coherent geometric strain softening can take place, then adiabatic strain softening is a requirement for the formation of shear bands by classical modes of plastic deformation (e.g. the motion of dislocation-like structures). However, if there were found to be evidence of a self-perpetuating (strain softening) mechanism which is activated only by the shear component of stress (i.e. a shear "fracture" mechanism) which for its initiation is only dependent on the elastic stress field, then this would explain the occurrence of localized shear zones in creep. Indeed, just such a mechanism has been proposed for spherulitic polyethylene [9, 10]. In this instance the "yield stress" of the polymer would be determined not by the plastic deformation of crystalline material but rather by the intrinsic shear fracture mechanism. In the specific case of polyethylene that mechanism is the cavitation of non-crystalline material or cavitation at the amorphous/crystalline boundary. Such a process would depend upon the distribution and effective modulus of non-crystalline material, molecular architecture, the number of tie molecules and degree of crystallinity (in the absence of effects from additives). If, say, under the given conditions of a test, the stress for cavitation initiated by shear, is less than that for homogeneous plastic flow caused by stress-activated rate processes, then "yield" will be initiated by cavitation. Support for such a mechanistic deformation transition is suggested in work involving uniaxial tensile tests on polypropylene [13]. Liu and Harrison [13] showed that as test rate increased the cold drawing of the polymer transformed into a mechanism involving "shear-banding" with an increase in sample volume caused by cavitation. Most importantly Liu and Harrison observed that this shear mechanism is essentially isothermal in nature and initiates below the elongation at which the first inflection in the load-elongation curve occurs in uniaxial tensile tests. Liu and Harrison also showed that the pre-drawing deformation in polypropylene is unaffected by a water environment. This would suggest that the transition

Figure 14 Broken-open BFF appear similar to shear bands in metals.

from homogeneous yield to "shear-banding with cavitation" is a transition from one isothermal mechanism to another. The deformation transition is thus mechanistic in nature and may therefore occur in creep. It is also of interest in this case to note that in the creep test of Lustiger and Cornieliusen [5] shear zones only formed in an agressive environment but did not form in air. The cavitation resistance of noncrystalline material would, in this instance, be lowered allowing the shear fracture (by micro cavitation) mechanism to compete with crazing. However, in our studies we have also found evidence of shear deformation zones in creep tests in a non-aggressive environment. The practical significance of the above is that if non-crystalline material can be designed to resist cavitation then the shear mechanism could be inhibited. In the case of polypropylene mentioned above, one could retain and improve the sub-yield cohesive properties at higher deformation rates. The inhibition of this mechanism may also influence failure in creep and fatigue.

The presence of additives and the different crystalline structure of materials used in the present study complicates the issue, however, Fig. 14 shows the broken open fracture surface of a BFF formed in the later stages of creep fracture. This is very similar to the fracture surfaces of shear-bands in metals. The current programme includes looking at such failure surfaces with energy dispersive analysis using X-rays (EDAX) and other structure sensitive techniques to determine if the particulate additives interact with the failure process. It is implicit in the above that geometrical considerations are'important in determining the mode of fracture.

4. Conclusions

The fatigue process in polyethylene is shown to be more complex than has previously been alluded to in the literature with a three-dimensional structure underlying what have previously been taken to be solid coherent fracture surfaces.

Bending the fracture surfaces thus reveals more information about the nature of fracture than would otherwise be apparent. (Similar findings have been reported previously when the plane strain fracture surfaces of polyethylene are bent [9].)

Creep and fatigue modes of fracture have been shown to have much in common providing support for the previously found correlation between creep and fatigue lifetimes [1].

Support is given to the reported micro-mechanisms of fatigue as proposed by Zhou and Brown [6] in particular respect of damage to the CLF during the unloading cycle.

Elucidation of the fracture process has thus allowed us to define areas of work and possible explanations of the observed failure modes (which are being pursued). In the process, a number of questions have already been raised as to the nature of fundamental mechanisms of deformation in polyethylene.

It is not clear (as yet) from any of our published or unpublished work involving scanning, transmission, or various types of optical microscopy, whether or not cavitation is involved as a precursor to the formation of the craze-like form at the crack tip, We have superficial evidence of competing mechanisms of craze formation, growth and breakdown, which include structure-dependent microcracking and micro-shearbanding.

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