JMEPEG (2012) 21:1468–1473
DOI: 10.1007/s11665-011-0024-z


1059-9495/$19.00

# Fracture Surface Analysis in HDPE Pipe Material Fatigued at Different Temperatures and Loading Frequencies

***Zafarullah Khan***

*(Submitted November 23, 2010; in revised form July 12, 2011)*

**Effect of temperature and loading frequency on the fatigue fracture process in high-density polyethylene (HDPE) pipe material has been investigated in this study via optical and scanning electron microscopy. Fatigue tests were performed using rectangular coupons obtained by slitting and flattening 50-mm-wide ring sections from 4-inch schedule 80 HDPE pipes. The flattening was carried out in a specially designed compression fixture at a temperature of 105 °C. Fatigue tests were conducted at temperatures of 0, 23, and 40 °C and loading frequencies of 0.1, 1, and 50 Hz. Fracture surface examinations reveal that the fatigue crack-growth process at all the test temperatures and loading frequencies involved mechanisms of shear yielding and crazing. Crack growth via crazing was found to be the dominant mechanism at higher temperature of 40 °C, while at 0 °C, a small amount of initial shear yielding precede the crazing process. Filler material particles contained in the HDPE pipe material play an important role of stress concentrators and help in micro-void nucleation, which promotes crack growth via crazing. The fatigue resistance of HDPE may thus be improved by addressing the stress concentration effect of filler particles.**

**Keywords** fatigue fracture, HDPE, temperature and frequency effects

## 1. Introduction

High-density polyethylene (HDPE) is gaining significant market share in the production of pipes used in natural gas and water transport/distribution networks. During service, these pipes are subjected to cyclic pressure fluctuations and experience failure by fatigue. In many applications, the pipes may also be exposed to non-ambient temperatures and various cyclic frequencies. To ensure reliable performance of the HDPE pipes, it is important to analyze their resistance to crack growth under cyclic loading at different temperatures and cyclic frequencies. While a considerable amount of research has been undertaken to study the rapid crack growth in HDPE (Ref 1-6), the issue of fatigue crack growth (FCG) has not yet been addressed adequately.

A number of studies have been conducted, which address the slow crack growth and FCG in many engineering plastics including HDPE (Ref 7-23). A general finding of these studies concludes that under stress states where the maximum principal stress is tensile, polymers may at the beginning of the deformation exhibit only a small amount or even no macroscopic inelastic deformation because of shear yielding. The initial inelastic deformation then switches to crazing. Crazing occurs as a two-step process; first, the highly stressed region decays into voids (creating craze gap) and initiates localized necking and micro-fibrils formation, which bridges the craze gap. Second, deterioration of the load-bearing capability of the craze fibrils leads to fracturing of the craze zone and propagation of the crack tip to the fore boundary of the craze zone. The crack is arrested, and waits for the next craze zone development and propagates its tip by once again fracturing the craze zone. The fracture thus occurs by a repetitive and sequential process of crack tip inelastic deformation (characterized by micro-void formation), craze zone development, craze-zone fracture, crack formation, and eventual crack growth to failure. Craze-zone fracture may occur by scission of the craze fibrils or by a pull-out failure of highly oriented chains in the craze zone. Fibril scission is favored at lower temperatures as well as at high strain rate (high frequencies), while chain pull out is expected at high temperatures and low strain rates (low frequencies).

**Zafarullah Khan**, Mechanical Engineering Department, King Fahd University of Petroleum & Minerals, Dhahran 31261, Saudi Arabia. Contact e-mail: zukhan@kfupm.edu.sa.

The slow crack-growth behaviors of HDPE and ethylene-hexene copolymer were compared. The slow crack-growth rate in the copolymer is about $10^2$-$10^3$ times slower than for the homopolymer. The kinetics of slow crack growth, the morphology of the damage zone, the stress-strain behavior, and the temperature dependence of damage rate were compared. The results suggest that the major effect of the butyl branches is to decrease the rate of disentanglement which governs the process of slow crack growth (Ref 9). The rates of initiation and growth of cracks in linear HDPE with different molecular weights were observed in single-edge-notched (SEN) tensile specimens under plane strain condition as a function of applied stress, notch depth, and temperature. The effect of $-M_w$ on the fast-fracture strength at low temperature and the relationship to tie-molecules have also been investigated. Quantitative relationships between the concentration of tie-molecules and the fracture behavior had been obtained previously (Ref 10). In a study of brittle slow crack growth in polyethylene, it was found that crazes formed at the crack tip, although secondary crazes were also evident. Multiple crack-arrest lines were also evident, suggesting a stick-slip mechanism under static load (Ref 24).

Formation and growth of the crack tip damage zone during slow stepwise crack propagation in polyethylene resins were studied experimentally. It was shown that improved fracture resistance correlated with development of an epsilon-shaped damage zone that consisted of the central craze and an accompanying pair of hinge shear zones of comparable length. The shear zones emanated from the crack tip immediately above and below the central craze where the highly stretched material formed a membrane that separated the crack tip from the cavitated material in the craze (Ref 18). FCG-accelerated tests were conducted on single-edge notch (SEN) specimens at the temperatures ranging from −10 to 70 °C and at frequencies ranging from 0.1 to 50 Hz. The crack-growth resistance was found to decrease with increasing test temperature and decreasing frequency (Ref 25).

Fatigue tests were carried on circumferentially notched bars (CNBs) of HDPE under controlled stress intensity (Ref 7). Those authors report that at $K^{max} < 0.25$ MPa $m^{0.5}$, crack propagation proceeds through a continuous stretching and breaking of micro fibrils in the localized craze at the crack tip. However, at larger $K^{max}$, the crack tip was seen to successively jump across the extended crazed zone where coarse fibrils formed from nucleated micro-voids. FCG in HDPE has been shown to occur with an accompanying layer of damage ahead of the crack tip. The overall crack-growth process reportedly involves initial crack growth at an accelerated rate, constant crack-growth rate, and re-acceleration of crack-growth rates to final failure (Ref 26). Within the first two regions, a "brittle" behavior is observed, while, in the third region, the cark growth is reported to occur in a "ductile" manner. It was proposed that two damage mechanisms of shear yielding and formation of fibrillated voids were responsible for HDPE failure. Another investigation also recognized that in HDPE the crack is indeed preceded by a layer of damaged material and identified a three-stage FCG process (Ref 27). Those authors report that a single craze-like active zone precedes the crack during the initial crack acceleration and a circular-shaped active plastic zone is responsible for crack deceleration, while the final failure occurs by crack re-acceleration through an elongated damage zone accompanied by a large-scale yielding. HDPE pipes tested at elevated temperatures under both the constant load and cyclic loading conditions revealed that the cracks were mostly initiated from the larger (>50 μm) filler material particles and grew in a discontinuous manner until final failure (Ref 28).

Despite the study thus far undertaken on investigating fatigue failure in HDPE, the crack-growth process, especially under non-ambient conditions is still a poorly understood phenomenon. The present study reports the findings of a study aimed at providing an understanding of FCG process in HDPE at different temperatures and loading frequencies.

## 2. Experimental Procedure

The flat plate SEN fatigue test specimens were obtained by flattening commercially procured 4-inch (100-mm) schedule 80

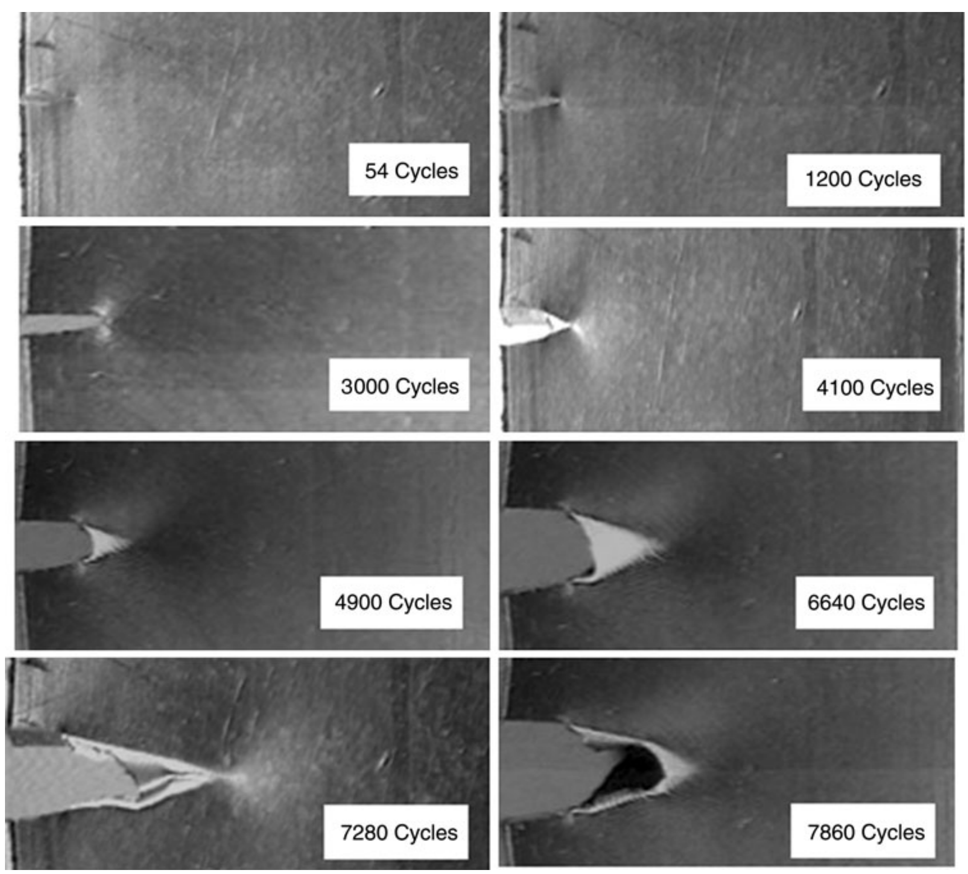


**Fig. 1** Macroscopic views of the development of craze zone and craze zone membrane in HDPE sample fatigued at 23 °C and at test frequency of 1 Hz

HDPE pipes. 50-mm-wide rings were cut from the pipes and slit into two equal c-shaped sections. The c-sections were heated at 105 °C in an electric oven for 20 min and flattened using a specially designed compression fixture. 40 mm × 180 mm × 9.4 mm fatigue test specimens were machined from the flattened c-sections. A sharp razor blade was employed to introduce a sharp through-thickness v-shaped starter notch of 1-mm depth at one edge of the test specimen.

A ±100 kN Instron® material testing machine was used to conduct fatigue tests. All the tests were carried out in load-control mode using sinusoidal load cycling at the frequencies of 0.1, 1, and 50 Hz and at temperatures of 0, 23, and 40 °C. A stress ratio of $R = 0.2$ and two stress levels ($\Delta\sigma$) of 13.3 and 11 MPa were used for the FCG tests. The non-ambient temperature tests were carried out by enclosing the test specimens in Plexiglas chambers. One chamber lined with heating mats maintained the test specimens at the uniform temperature of 40 °C. The 0 °C tests were carried out using another chamber in which a cryogenic fluid was circulated through copper tubing to maintain the specimens at temperature of 0 °C. Both chambers were well insulated to ensure a uniform temperature within ±1 °C for the entire duration of each test. Each chamber had a 30 × 60 mm$^2$ glass window to allow optical observation of the fatigue crack process. After fatigue testing, the fracture surface morphology of the failed specimen was examined using a Joel scanning electron microscope.

## 3. Results and Discussion

### 3.1 Macroscopic Crack-growth Observations

Similar to many other polymers, the progress of fatigue fracture in HDPE also takes place with the formation and rupture through initial shear yielding and subsequent crazing process. The series of optical photographs captured during the fatigue testing (see Fig. 1) provides evidence of such a fracture process in a HDPE sample fatigued at a temperature of 23 °C and a test frequency of 1 Hz. The first fully developed craze zone appears at around 3000 cycles. A thick membrane, which consists of highly stretched fibrils, can be seen developing at the crack tip. This membrane keeps the two fracture surfaces connected until the craze zone fractures develop to complete the crack-growth step. As it is evident from examination of Fig. 1, the crack growth occurs by formation of craze zone and craze zone rupture through dismemberment of the stretched fibrils of the stretched craze zone membrane.

### 3.2 SEM Fracture Surface Analysis

The SEM fractographs in Fig. 2(a) and (b) show the fracture surface morphology of a HDPE specimen fatigued at 23 °C at a test frequency of 1 Hz. These fractographs were taken at different positions along the crack path.

The fractograph shown in Fig. 2(a) displays an area of approximately 4 mm from the starter notch, with the fracture surface providing clear evidence of a crazing process originating from multiple sites. The fracture surface examination suggests that the crazing process proceeds with crack arrests during the intermittent craze zone fibril formation and fracture. The fractograph (Fig. 2b) which is taken at a distance of approximately 8 mm from the machined notch shows small filler material particles at the bottom of the micro-voids

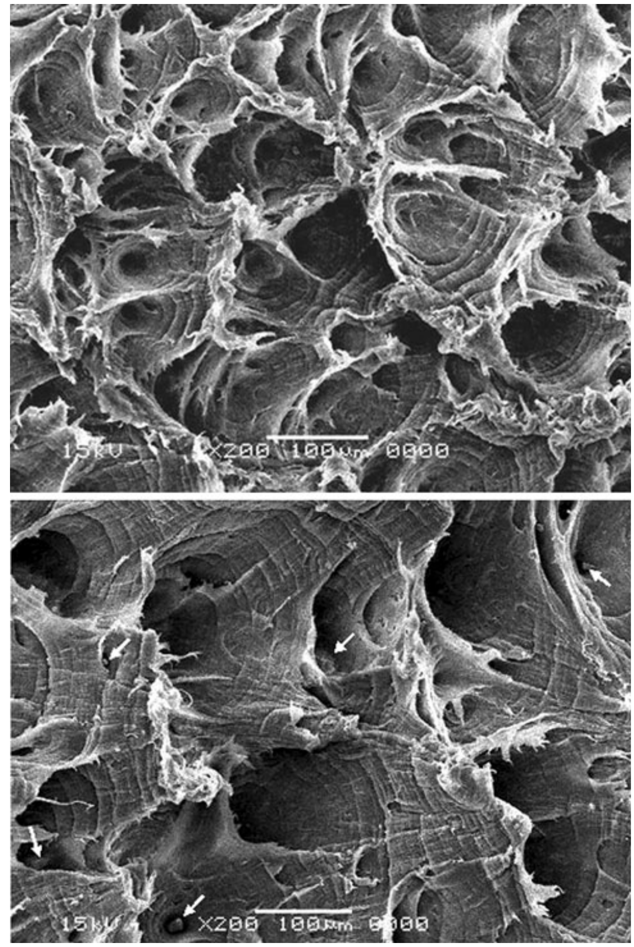


**Fig. 2** SEM fractograph showing the fatigue fracture process in HDPE sample fatigued at 23 °C and at a frequency of 1 Hz: (a) 4 mm from the notch, and (b) 8 mm from the notch. The arrows in (b) point at the filler material particles

suggesting that these may have in fact acted as hard and brittle stress raiser particles within a ductile matrix and promoted micro-void formation. The filler materials (such as $CaCO_3$) are commonly added to HDPE pipe material to enhance physico-chemical properties by improving crosslinking and inhibit swelling (Ref 29, 30). The role of such filler material particles in the fracture have also been reported by other researchers (Ref 21). Figure 2(b) also show crack-arrest markings on the fracture surface, which indicate that the crack growth at this stage occurs through a fully developed crazing process.

A change in the test frequency from 1 to 50 Hz at the temperature of 23 °C resulted generally in similar two stages in the crack-growth process. The region immediately adjacent to machined notch exhibited a shear yielding damage marked by cellular structure and matted down shear regions (Fig. 3a). However, at this frequency, it appears that more material has been drawn into the plastic zone ahead of the crack tip than that occurs at 1 Hz. At 2 mm from the machined notch, the fracture surface shows clear crack-arrest markings indicating an early onset of the fracture process through micro-void formation, multiple crazings, and fibril scission (Fig. 3b). The fracture surface, however, shows some areas especially at the edges of the micro-voids, where the drawn material may have experienced excessive temperature rise, which may have resulted in localized melting of the fibrils.

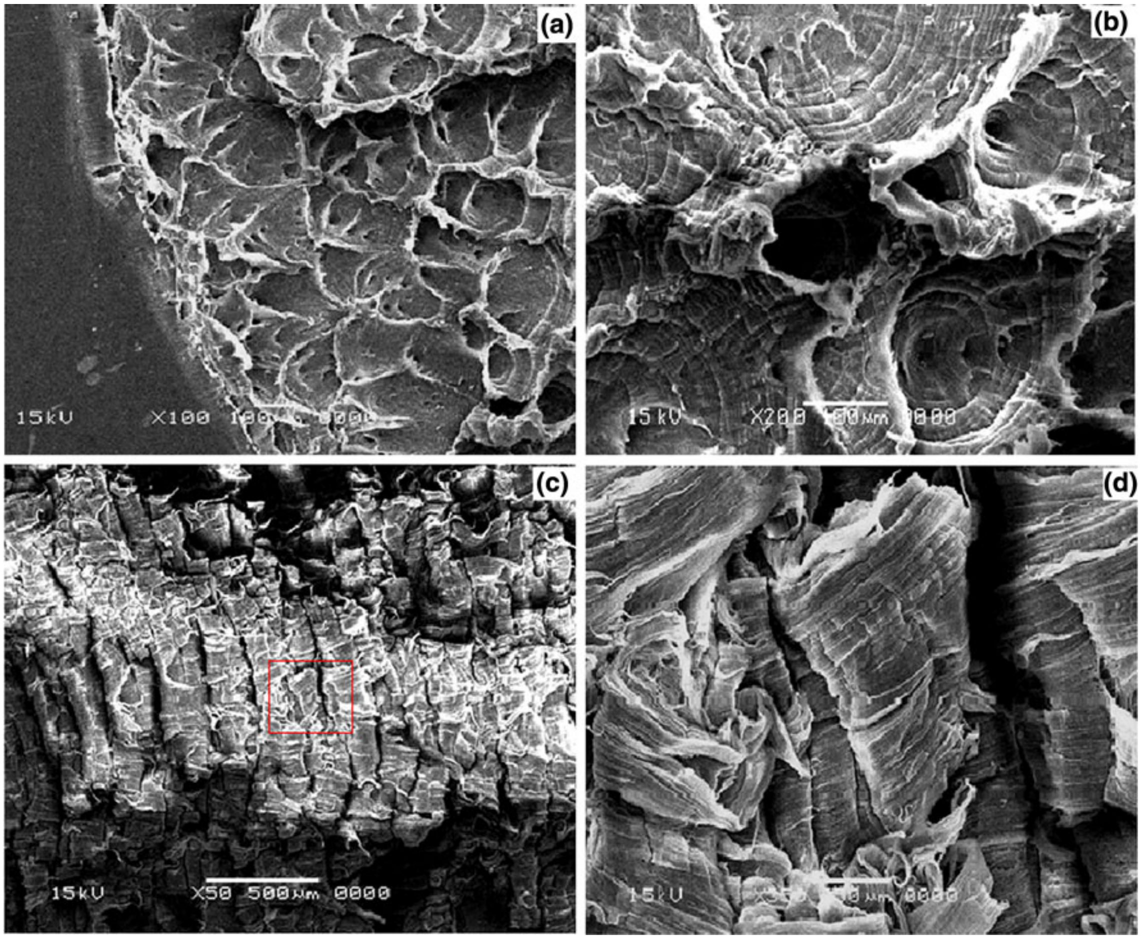


**Fig. 3** Series of SEM fractographs showing the fracture surfaces in HDPE sample fatigued at 23 °C and 50 Hz: (a) Shear yielding at the notch, (b) at 2 mm, (c) at 5 mm, and (d) higher magnification view of the boxed area of (c)

Such melting has also been reported for the HDPE by Brough et al. (Ref 31) in their study of fast fracture of HDPE under impact loading. Melting of the fibril material is a result of the elevation of the temperature due to crack-tip hysteresis heating expected at high-frequency cyclic loading. At a distance of approximately 5 mm from the notch (Fig. 3c), the fracture surface shows a craze deformation zone where crack arrest seems to have resulted in secondary cracking. It is expected that thermal softening of the material would result in highly deformed material in the damaged zone and would be unable to transfer the load from one deformation zone to the next, which would lead to concentration of the stress locally at the previous crack-arrest zone and cause the discontinuity to form. A clear evidence of thermal softening and melting of the polymer fibrils could be seen in the higher magnification fractograph (Fig. 3d) of the boxed area of Fig. 3(c). The figure shows several areas where heavily drawn fibrils have fused together as a result of such thermal softening and melting.

When the test frequency is changed to 0.1 Hz, the fracture surface appears somewhat different than what was observed at the other two frequencies of 1 and 50 Hz. A comparatively flatter surface spans most of the fatigue fracture zone (Fig. 4a). The crack growth appears to have involved a mixed shear and crazing process. The heavily drawn material seen on the fracture surface and less pronounced cavitation suggests that the crack growth at the frequency of 0.1 Hz may have progressed predominantly by a shear yielding process rather than by crazing (Fig. 4b). This observation is supported by the absence of extensive crack-arrest markings on the surface as were observed at the other two frequencies of 1 and 50 Hz.

At 0 °C and a test frequency of 1 Hz the fracture surface has features representing a much less ductile fracture than observed at 23 °C. Figure 5(a) represents an area approximately 4 mm from the notch. A rather flat-faceted fracture surface morphology points at the fact that the material assumes a somewhat brittle character at the lower temperature of 0 °C.

The crack growth at 0 °C, however, still occurs by a mixed-mode shear yielding and crazing process as evidenced from extensive cavitation and crack-arrest markings on the fracture surface seen in Fig. 5(b). The role of the second-phase additive brittle particle in providing the stress concentration sites for micro-void formation can also be clearly seen in this figure.

The samples tested at 40 °C and a test frequency of 1 Hz exhibit rather gross yielding of the bulk material as shown in Fig. 6. The test sample has experienced extensive necking at a test temperature of 40 °C. The fracture surface is rather flat and featureless, and provides no evidence that the fracture process has involved the typical shear yielding or crazing. A magnified view of the region adjacent to the machined notch reveals a

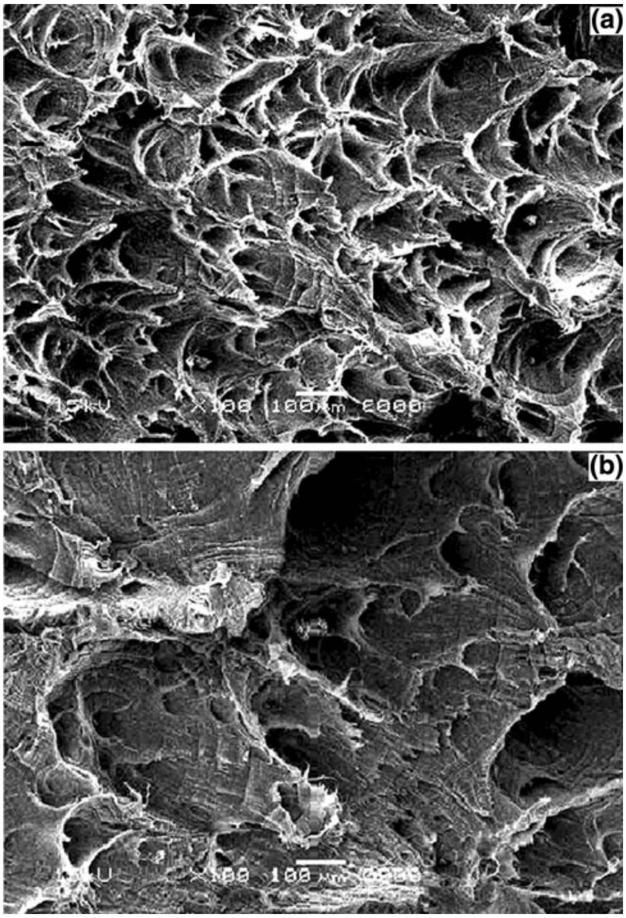


**Fig. 4** SEM fractographs showing the fatigue fracture process in HDPE sample fatigued at 23 °C and a test frequency of 0.1 Hz. (a) Shear yielding and crazing at the notch and (b) 8 mm form the starter notch

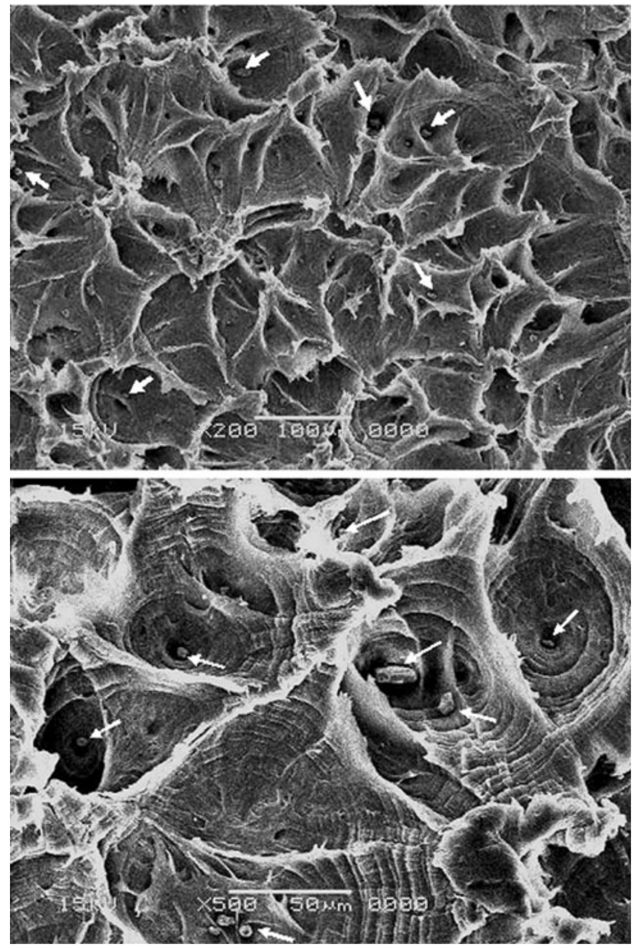

**Fig. 5** SEM fractographs showing the fatigue fracture process in HDPE sample fatigued at 0 °C and a test frequency of 1 Hz: (a) 4 mm from the notch, and (b) cavitation and crack arrest at 8 mm. The arrows point at the filler material particles

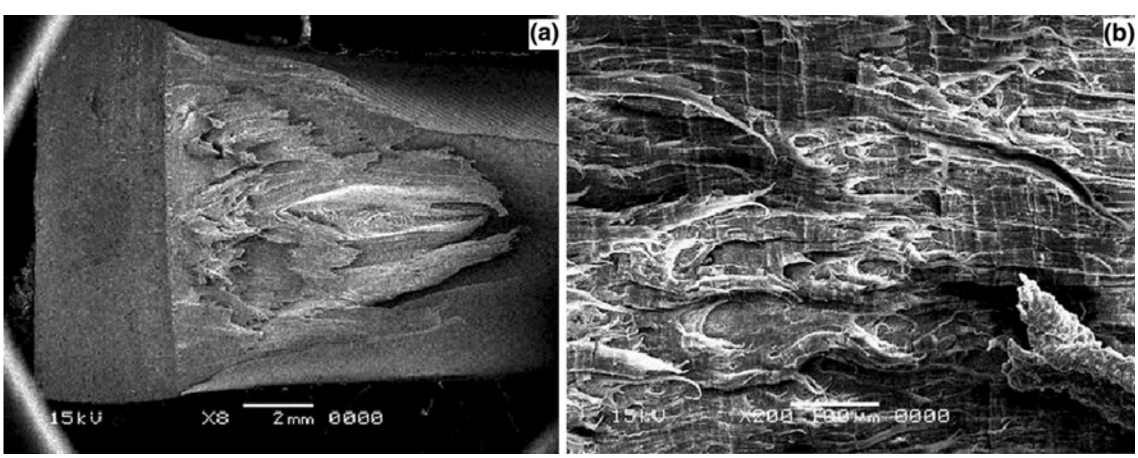


**Fig. 6** SEM fractographs showing the HDPE sample fatigued at 40 °C and a frequency of 1 Hz: (a) Gross yielding and (b) magnified view of the fracture surface near machined notch

very rapid crack-growth process with a fewer localized crack arrests during the growth process.

At higher frequency of 50 Hz, the fracture surface of a specimen fatigued at 40 °C provides clear evidence that the crack growth predominantly involves a crazing process (Fig. 7). It is also interesting to notice that, at this high-frequency hysteresis, crack tip heating has caused localized fibril softening and melting as evident from Fig. 7.


**Fig. 7** SEM fractograph showing the HDPE sample fatigued at 40 °C and a frequency of 50 Hz

## 4. Conclusions

Fatigue fracture process in HDPE pipe material has been examined via optical and scanning electron microscopy. Fracture surface examinations reveal that the overall fatigue fracture process in HDPE pipe material involves initial crack growth by shear yielding, followed by crack growth via crazing. Crack growth via crazing dominates at a higher temperature of 40 °C, while at 0 °C, a small amount of initial shear yielding precede the crazing process. The additive material particles present in HDPE pipe material plays a significant role in the craze formation. These hard particles act as stress concentrators, helping in the void formation at multiple sites and promotion of the crazing process.

## Acknowledgment

The author wishes to gratefully acknowledge the support for this research provided by the King Fahd University of Petroleum & Minerals, Dhahran, Saudi Arabia.

## References

1. R.K. Krishnaswamy, M.J. Lamborn, A.M. Sukhadia, D.F. Register, and P.L. Maeger, Rapid Crack Propagation Failures in HDPE Pipes: Structure-Property Investigations, *Polym. Eng. Sci.*, 2006, **46**(10), p 1358–1362
2. G. Palermo, W.J. Michie, Jr., and D. Chang, *Rapid Crack Propagation Increasingly Important in Gas Application, A Status Report*, Vol. 235(12), The Dow Chemical Company, Midland, MI, 2008
3. P. Yayla and P.S. Leevers, Rapid Crack Propagation in Pressurised Plastic Pipe—II. Critical Pressures for Polyethylene Pipe, *Eng. Fract. Mech.*, 1992, **42**(4), p 675–682
4. W. Zhou, E. Golovin, M. O'Leary, and A. Chudnovsky, *Proceedings of the 68th Annual Technical Conference of the Society of Plastics Engineers 2010*, 16-20 May 2010 (Orlando, FL), Vol. 1, p 171–175
5. C.J. Greenshields and P.S. Leevers, Fast Brittle Fracture of Plastic Pipes. Part 1: Water Pressurised, *Plast. Rubber Compos. Process. Appl.*, 1997, **26**(9), p 387–393
6. G.P. Venizelos, C.J. Greenshields, A. Ivankovic, and P.S. Leevers, Fast Brittle Fracture of Plastic Pipes, Part 2—Gas Pressurized, *Plast. Rubber Compos. Process. Appl.*, 1997, **26**(9), p 394–403
7. V. Favier, T. Giroud, E. Strijko, J.M. Hiver, C. G'Sell, S. Hellinckx, and A. Goldberg, Slow Crack Propagation in Polyethylene Under Fatigue at Controlled Stress Intensity, *Polymer*, 2002, **43**, p 1375–1382
8. P.A. O'Connell, R.A. Duckett, and I.M. Ward, Brittle-Ductile Transitions in Polyethylene, *Polym. Eng. Sci.*, 2002, **42**(7), p 1493–1508
9. X. Lu, X. Wang, and N. Brown, Slow Fracture in a Homopolymer and Copolymer of Polyethylene, *J. Mater. Sci.*, 1988, **23**(2), p 643–648
10. Y.L. Huang and N. Brown, The Effect of Molecular Weight on Slow Crack Growth in Linear Polyethylene Homopolymers, *J. Mater. Sci.*, 1988, **23**(10), p 3648–3655
11. K. Kitao, Effect of Entanglement on the Brittle-Ductile Transition in Polyethylene, *Polym. Eng. Sci.*, 2001, **41**(7), p 1146–1155
12. S.K. Phua, C.C. Lawrence, and R. Potter, Fractographic Study of High-Density Polyethylene Pipe, *J. Mater. Sci.*, 1998, **33**, p 1699–1702
13. H.S. Kim and X.M. Wang, Temperature and Frequency Effects on Fatigue Crack Growth of uPVC, *J. Mater. Sci.*, 1994, **29**, p 3209–3214
14. N. Merah, F. Saghir, Z. Khan, and A. Bazoune, Variation of Mechanical Properties of HDPE Pipe Material with Temperature, *J. Plast. Rubber Compos. Macromol. Eng.*, 2006, **35**(5), p 226–230
15. C.B. Bucknall and P. Dumpleton, Effects of Loading History on Fatigue Crack Growth in High Density Polyethylene and Toughened Poly(Methyl Methacrylate), *Polym. Eng. Sci.*, 1987, **27**(2), p 110–115
16. X. Lu, R. Qian, and N. Brown, Discontinuous Crack Growth in Polyethylene Under a Constant Load, *J. Mater. Sci.*, 1991, **26**(4), p 917–924
17. A. Dasari and R.D.K. Misra, Microscopic Aspects of Surface Deformation and Fracture of High Density Polyethylene, *Mater. Sci. Eng. A*, 2004, **367**, p 248–260
18. M. Parson, A. Stepanov, A. Hiltner, and E. Baer, The Damage Zone Ahead of the Arrested Crack in Polyethylene Resins, *J. Mater. Sci.*, 2001, **36**, p 5747–5755
19. H.S. Kim and X.M. Wang, Temperature and Frequency Effects on Fatigue Crack Growth in Acrylonitrile-Butadiene-Styrene (ABS), *J. Appl. Polym. Sci.*, 1995, **57**, p 811–817
20. D. Ferrer-Bals, M.L. Maspoch, and Y. Mai, Fracture Behavior of Polypropylene Films at Different Temperatures: Fractography and Deformation Mechanisms Studied by SEM, *Polymer*, 2002, **43**(10), p 3083–3091
21. L. Engel, H. Klingele, G.W. Ehrhenstein, and H. Schafer, *An Atlas of Polymer Damage: Surface Examination by Scanning Electron Microscope*, Wolfe Publishing, London, 1981
22. R.W. Hertzberg and J.A. Manson, *Fatigue of Engineering Plastics*, Academic Press, New York, 1980
23. S.H. Beech and J.N. Mallinson, Slow Crack Growth Performance of Today's Plastics Pipeline Materials, *Plast. Rubber Compos. Process. Appl.*, 1998, **27**, p 418–423
24. A. Lustiger and R.D. Corneliussen, The Role of Crazes in the Crack Growth of Polyethylene, *J. Mater. Sci.*, 1987, **22**(7), p 2470–2476
25. N. Merah, Z. Khan, A. Bazoune, and F. Saghir, Temperature and Loading Frequency Effects on Fatigue Crack Growth in HDPE Pipe Material, *Arab. J. Sci. Eng.*, 2006, **31**(2C), p 19–30
26. M. Kasakevich, A. Moet, and A. Chudnovsky, A Crack Layer Approach to Fatigue Crack Propagation in High Density Polyethylene, *J. Appl. Polym. Sci.*, 1990, **39**(2), p 395–413
27. K. Sehanobish, A. Chudnovsky, and A. Moet, Crack Layer Analysis of Nonmonotonic Fatigue Crack Propagation in High Density Polyethylene, *Polymer*, 1987, **28**(8), p 1315–1320
28. A. Chudnovsky, K. Sehnobish, and S. Wu, Methodology for Durability Analysis of HDPE Pipe, *J. Press. Vessel Technol.*, 2000, **122**, p 152–155
29. F. Mijangos-Santiago and J.M. Dealy, Effect of Filler Content and Additives on the Extrudate Swell of Polyethylene Pipe Resin, *Polym. Eng. Sci.*, 1991, **31**(16), p 1176–1181
30. C. Vasile and M. Pascu, *Practical Guide to Polyethylene*, Smithers Rapra Press, Shrewsbury, 2008
31. I. Brough, R.N. Haward, and G. Healy, Scanning Electron Micrographs of High Density Polyethylene Fracture Surfaces, *Polymer*, 2004, **45**(10), p 3115–3123