The Effect of Pressure and Contaminants on Slow Crack Growth in a Butt Fusion in a Polyethylene Gas Pipe

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SYNOPSIS

The effect of welding pressure and surface contaminants on butt fusion in a commercial polyethylene gas pipe was studied by measuring the slow crack growth resistance of the joint. Two weld pressures, 1.61 and 0.31 MPa, were used. The contaminants Vaseline, graphite, and Teflon were coated on the surface. It was found that the high pressure produced a weld with much better slow crack growth resistance than that by low pressure. At high weld pressure, Vaseline and graphite showed a negligible effect on weld quality. Teflon greatly reduced the lifetime, especially at low weld pressure. Test results showed that sufficient pressure was necessary to squeeze out the thin skin layer that occurs at the weld interface, along with any contaminants. The butt fusion joints were always much weaker than the reference pipe. This result was attributed to the difference in both cooling rate and molecular orientation between the pipe and weld. © 1992 John Wiley & Sons, Inc.

1. INTRODUCTION

This investigation was initiated because the producer and user of the same gas pipe recommended different procedures for producing a butt weld. The only difference was in the pressure to produce the weld. This difference was obviously reflected in the shape of the weld bead as shown in Figure 1. The user could find no difference in the short-time tensile test between the two pressures, but found that the impact strength was better for the low pressure. The user also found that the low-pressure weld could be produced more consistently by their service personnel. The important question to be answered is whether the high or low pressure produced the weld with the longest lifetime as determined by the mechanism of slow crack growth. To eliminate the human factor with respect to the application of the pressure, an automatic computer-controlled butt fusion apparatus was used.

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An additional variable, the effect of surface contamination, was investigated. The effect of specific contaminants are of interest in their own right, but the study of the combined effects of pressure and surface contamination leads to a more fundamental understanding of the butt fusion process. This study also tried to answer the question of why the lifetime of the pipe is greater than that of the weld.

Atkinson and co-workers[1-3] studied the morphology of the weld as shown in Figure 2. They showed the importance of the "remnant skin" that occurs at the interface. The skin forms during the dwell time—the interval between the removal of the pipe from the hot plate and the time the pipes are butted together. The skin can be greatly altered by a contaminant. The degree of removal of the remnant skin during the application of the pressure significantly affects the quality of the weld.

Aoki et al.[4] studied the effect of weld pressure. They found that the impact energy of the weld decreased with increasing pressure and then reached a constant value. Using a notched tensile specimen to measure the lifetime under slow crack growth, they found that the lifetime reached a maximum value at an intermediate value of the butting pres-

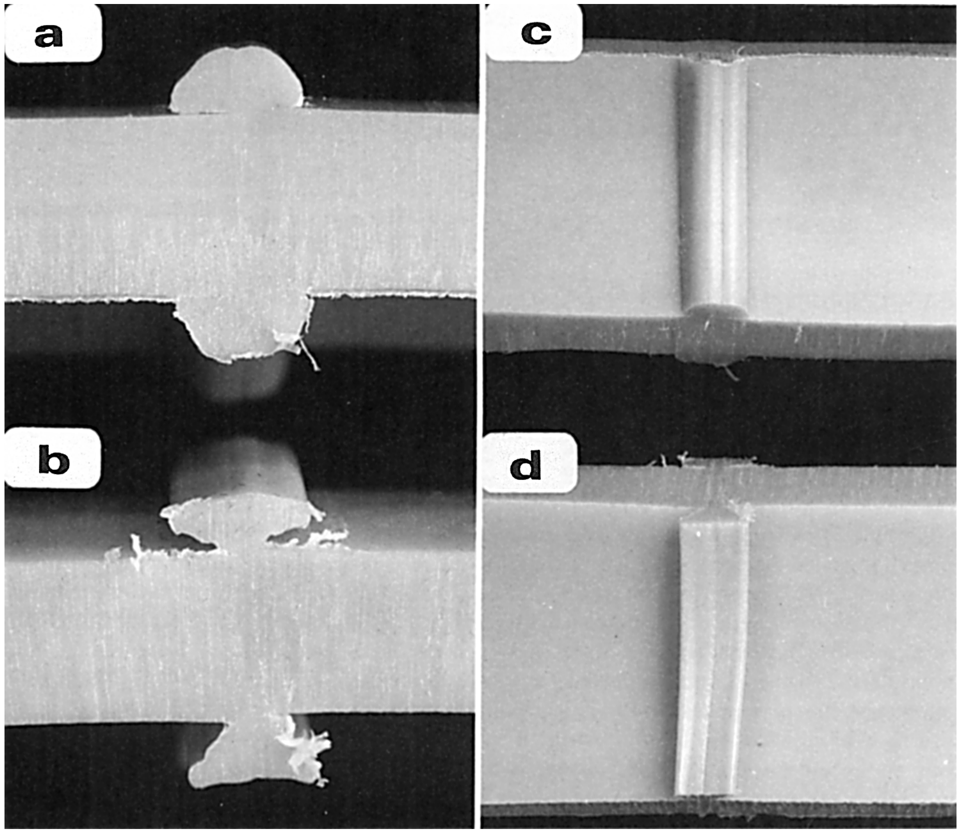

Figure 1 Shape of beads: (a,c) 1.61 MPa; (b,d) 0.31 MPa. (a) and (b) are sideview; (c) and (d) are the view of the inside surface of the weld joint.

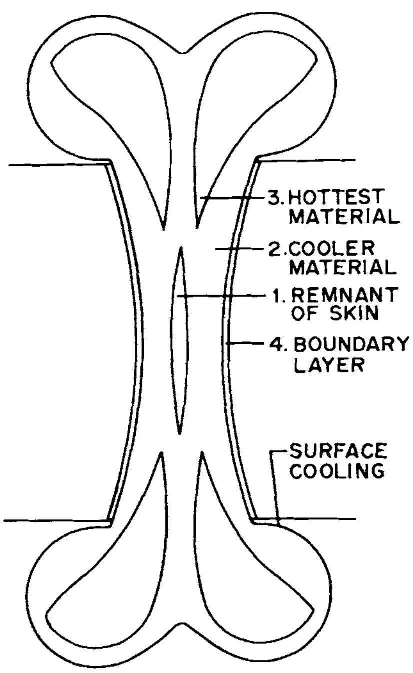

Figure 2 Schematic picture to show five zones in the weld joint of polyethylene (from Atkinson et al.[1-3]).

sure. The effect of pressure on lifetime could be explained as follows: At low pressure, the remnant skin is not fully removed so that the adhesion is not uniform throughout the interface. If the pressure is too high, the molecules are too well oriented in the plane of the interface so that slow crack growth occurs more readily. Why the impact energy, which is associated with rapid fracture, decreased with increasing weld pressure as found by the user and also in the Aoki investigation is not clear. The factors that affect rapid fracture strength are not the same as those for slow crack growth.

Pimputkar[5] explored the ranges of the welding parameters that produce good welds based on a short-time fracture test. The optimum range of pressures obviously depends on the temperature of the interface since the melt viscosity is temperature-dependent. In this study it is demonstrated that the higher pressure of 1.61 MPa gave a longer lifetime than did the 0.31 MPa pressure. This result applies only to the specific values of the other welding parameters that were used in this study.

This investigation along with others has shown that the lifetime of the weld is generally about one-half that of the pipe. Causes for this effect were also investigated.

2. EXPERIMENTAL

Tests were done on a 110 mm-diameter commercial gas pipe with wall thickness of 10.5 mm. It was made from an ethylene–octene copolymer with about 4.5 hexyl branches/1000 C. The MI was 0.3 g/10 min (ASTM 1238 2.16 Kg) and M_n = 33,000 and M_w = 128,000.

The butt welding was done with a BF3-type automatic butt fusion equipment manufacturer by Fusion Equipment. All the welding parameters were automatically controlled by a preprogrammed computer. The pipes were trimmed by the machine and were very carefully aligned. The pipe ends were heated for 90 s under a slight pressure against the hot plate whose surface temperature was 172°C. The heater was automatically removed and the ends of the pipe were brought forward with a fixed amount of travel to a constant pressure in a period of 2 s. The pipes were held at the constant pressure for 542 s, after which time the pipe was removed. The only variable was the pressure that was 1.61 or 0.31 MPa.

After trimming, the contaminants were applied to one-half of the surface of each pipe. The Vaseline was smeared lightly but uniformly on the surface. The graphite lock fluid and the Teflon dry lubricant were sprayed on the surface. The uncontaminated surfaces were used as a reference for evaluating the effect of each contaminant.

After removing the weld bead, the specimen shown in Figure 3 was milled from the pipe. A 3.5 mm-deep notch was made on the inside surface of the pipe. A fresh razor blade was used for each notch and was pressed into the pipe at a rate of 50 mμ/min. Side groves, 1 mm deep, were made with the razor blade to diminish the effect of the surface and to produce the same plane strain fracture that occurs in service. A low-power magnifier was used to identify the weld interface to place the notch in the plane of the weld interface. Notches were made in the pipe itself perpendicular or parallel to the longitudinal direction of the pipe to examine the effect of anisotropy in the pipe.

The notched specimens were exposed to a constant tensile stress that was sufficiently low in order to produce the brittle-type failure that is observed when gas pipes fail after a long time in service. Extensive experiments with the same resin by Lu and Brown[6] identified the conditions of temperature and stress that produce long-time brittle fracture.

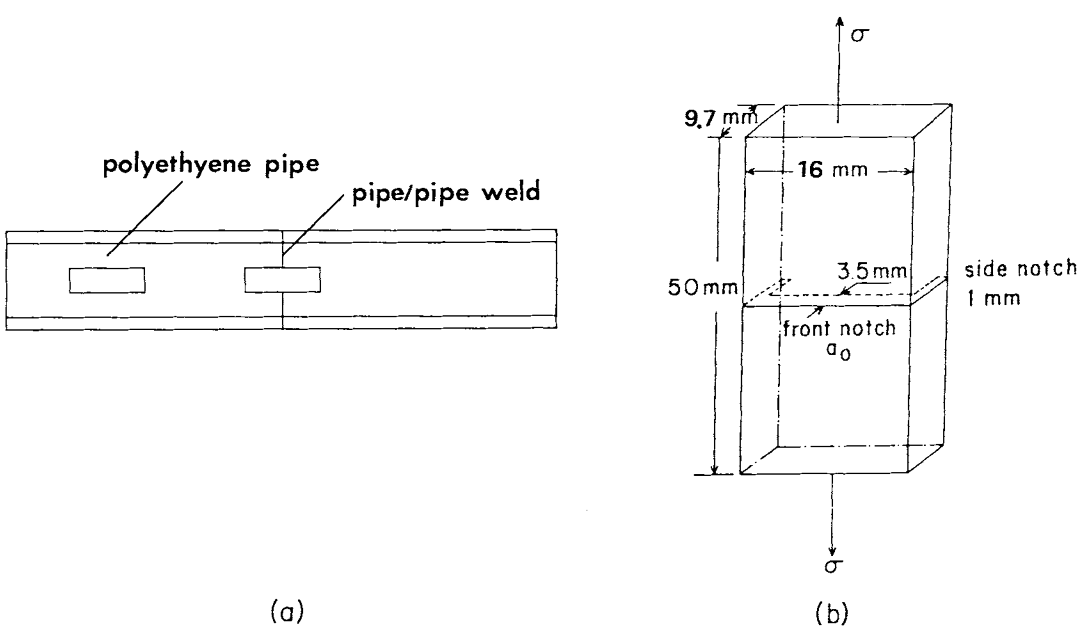

Figure 3 (a) Orientation of test specimens from weld joint and reference pipe; (b) specimen geometry.

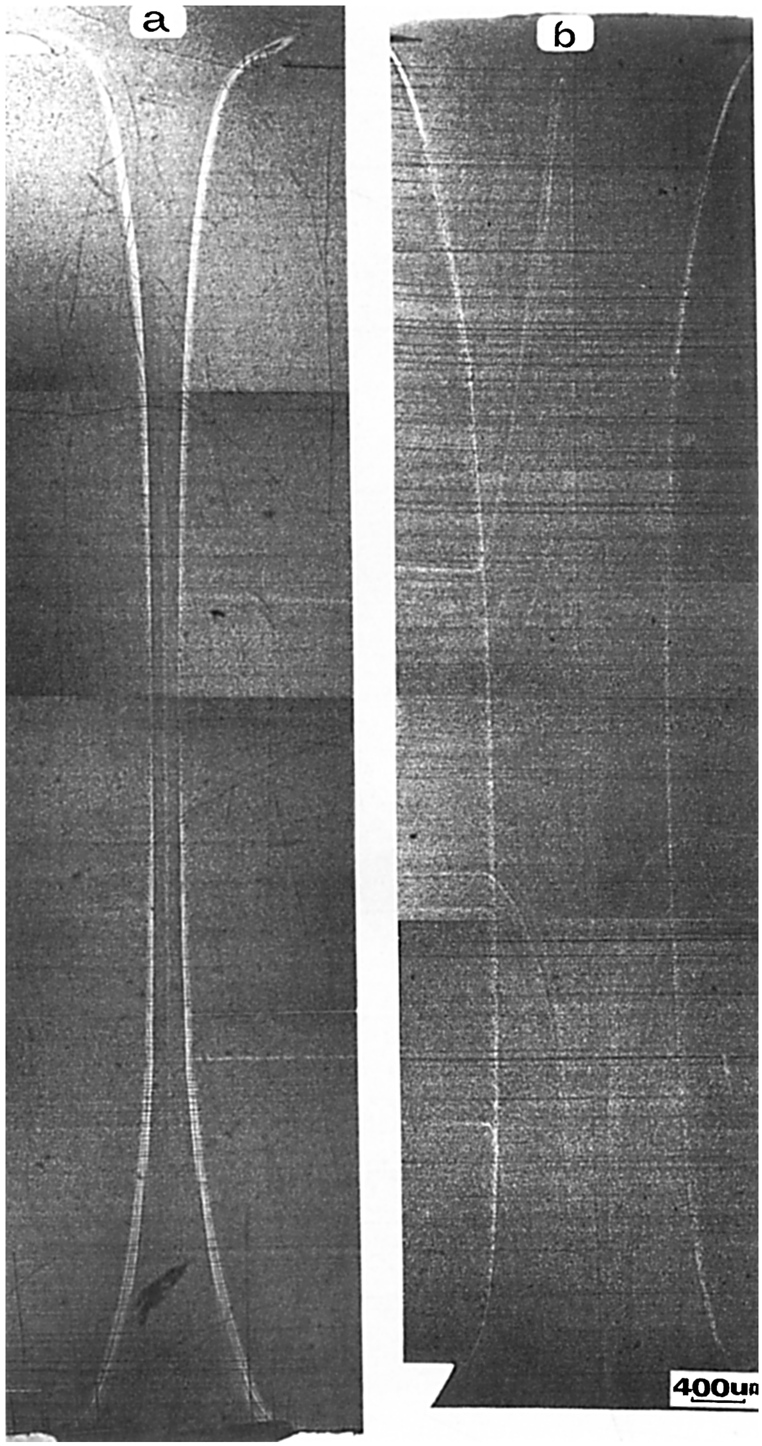

Figure 4 Butt fusion zone at two weld pressures: (a) 1.61 MPa; (b) 0.31 MPa.

The crack opening displacement (COD) was measured with an optical microscope having a filar eyepiece. Three to four duplicate tests were made for each set of conditions.

3. EXPERIMENTAL RESULTS

Figure 4 shows the fusion zone for 1.61 and 0.31 MPa pressure. The higher pressure extrudes more

Table I Effect of Weld Conditions on Lifetime (42°C, 4.5 MPa)

Weld Pressure (MPa)	Lifetime (min)	Average
1.61	3550, 2248, 2808 2938, 2079, 2119	2624 ± 22%
0.31	1301, 1032, 2186 1518, 2347, 1480	1664 ± 31%
Reference pipe notch ⊥ to *z*	5134, 5553	

of the molten zone so that its minimum thickness was 0.2 mm as compared to 1.39 mm for the lower pressure. The shape of the beads for each pressure is shown in Figure 1. Figure 4 shows flow lines. The flow lines at the interface indicates that the molecules are oriented in the plane of the interface and thus would lie in the plane of the notch.

Figure 5 shows COD vs. time for welding pressures of 1.61 and 0.31 MPa and for the reference pipe. The discontinuous crack growth is a feature that is often found during slow crack growth in gas pipes as shown by Lu and Brown.[7] Table I shows

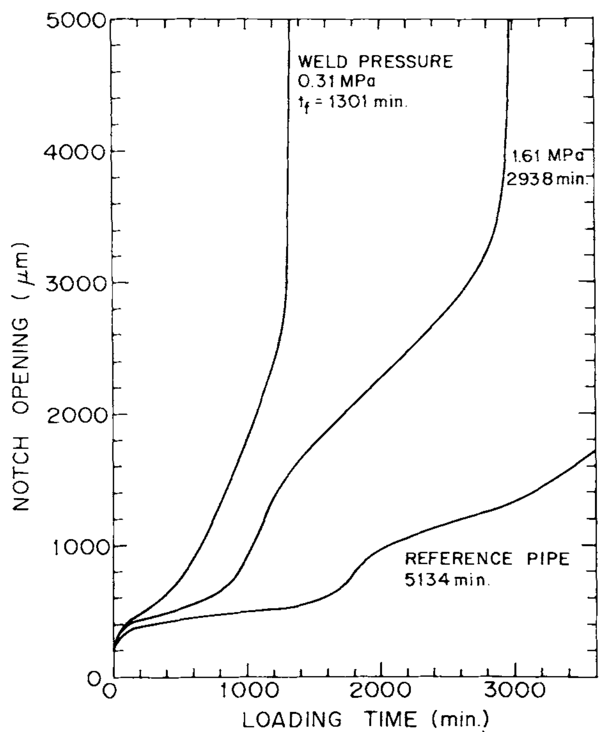

Figure 5 Notch opening vs. loading time for butt fusion joint and reference pipe specimen tested at 42°C and 4.5 MPa.

that the average lifetime at 1.61 MPa pressure is 2624 min, and for 0.31 MPa, 1644 min, with a lifetime of the 5344 min for the reference pipe. The scatter in the lifetime of the welded joint is greater than for the pipe. Many tests on the pipe in previous investigations give a scatter in lifetimes within ±15%.

Table II shows the effect of the contaminants. The graphite and Vaseline contaminants produced about the same life, which is not significantly different from the lifetime of an uncontaminated joint. Teflon sprayed on the surface significantly reduced the life by a factor of about 3 for a pressure of 1.61 MPa. For a pressure of 0.31 MPa, the Teflon produced a joint that was almost completely unbonded.

The micrograph in Figure 6 shows that the fracture surface contains the interface between the two molten zones. Figure 7 shows that for a pressure of 1.61 MPa the fractured surface, where slow crack growth occurs, is fully fibrillated as it is for fracture in the pipe. However, for a pressure of 0.31 MPa, there are regions that are not completely fibrillated. Figure 8 shows that the character of the fibrillation on the fractured surface of the weld at 1.61 MPa is

Table II Effect of Contamination on Lifetime (42°C, 4.5 MPa)

Contaminators	Lifetime (min)	Average
Vaseline[a]	3282, 2607, 3339	3076
Graphite[a]	2432, 3523, 1668, 3757	2845
Teflon[a]	577, 1470, 1959, 467	1118
Clean surface[a,*]	2823, 3930, 2947,	3233
Teflon[b]	4.8, 0.8, 0.8, 2	2.1

[a] Weld pressure, 1.61 MPa.
[b] Weld pressure, 0.31 MPa.
* The specimens were taken from the uncontaminated surface of the welding.

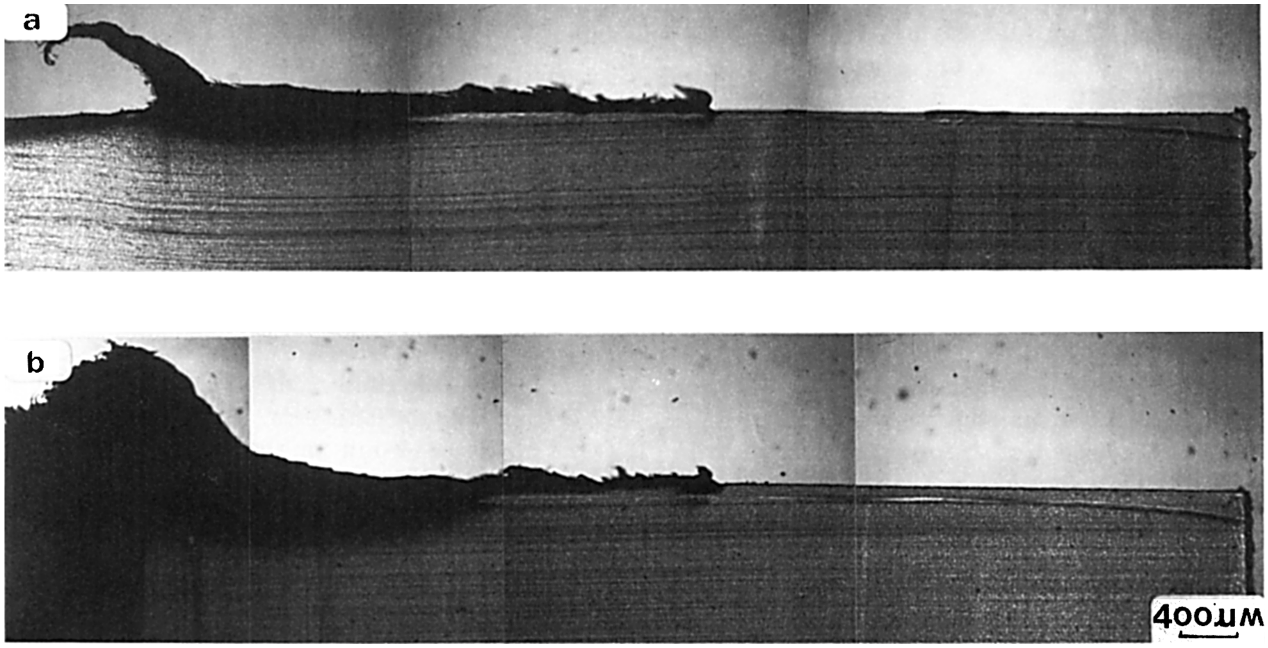

Figure 6 Side view of the fractured specimen welded at a pressure of 1.6 MPa and with a lifetime of 3550 min; (a) and (b) were sliced at different locations along specimen width direction. Fracture is shown in the center of the molten zone.

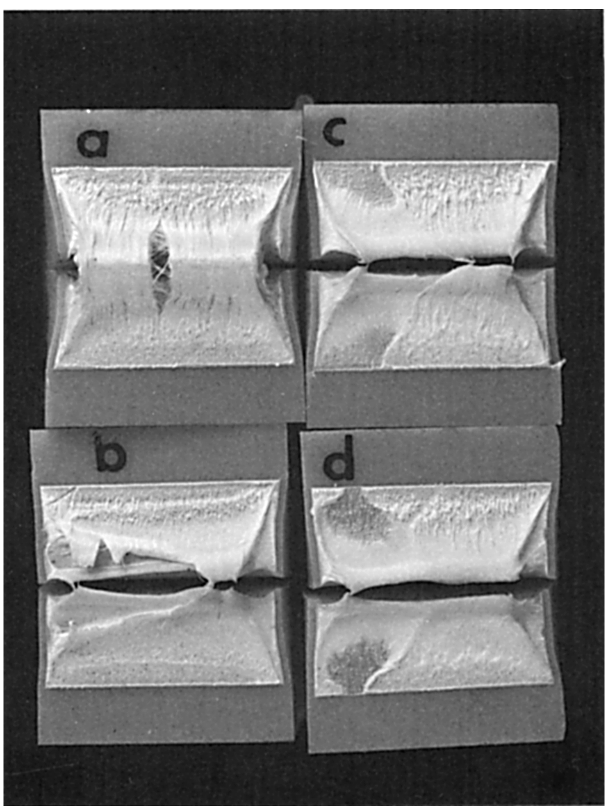

Figure 7 Fracture surface by optical microscopy: (a) pipe; (b) weld at 1.61 MPa; (c,d) weld at 0.31 MPa.

not appreciably different from that on the reference pipe. Figure 9 shows the unfibrillated regions that occur at a pressure of 0.31 MPa. Apparently, the lower pressure does not produce uniform contact between the molten surfaces since some regions are well fibrillated and others appear unbonded. The unbonded part is assumed to be related to the "remnant skin."

Figures 10 and 11 show that the morphology of the fractured surfaces with contaminants. Vaseline and graphite contaminants are not distinguishable from those on clean surfaces. The Teflon-contaminated surface under 1.61 MPa pressure showed uniform fibrillation, but the fibrillation is significantly less dense than those on a clean surface. Figure 12 shows that under a pressure of 0.31 MPa the Teflon-contaminated surface was completely unbonded in many areas.

To understand the difference in lifetimes between the pipe and the welded joint, the following experiments were performed: Notches were made parallel and perpendicular to the longitudinal axis of the pipe. Table III shows that when the fractured surface is perpendicular to the longitudinal axis the lifetime is greater by a factor of 1.5 compared to the parallel direction. Another factor to be considered is the effect of cooling rate on lifetime. Table III shows that

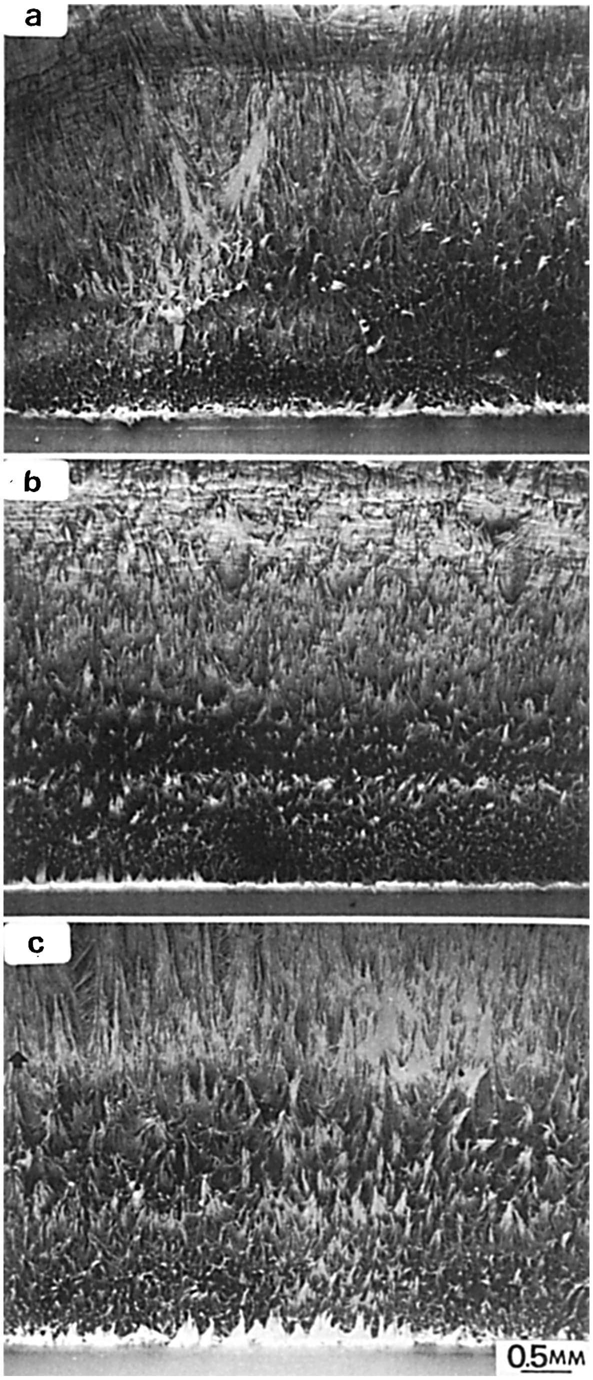

Figure 8 Fibrils on fractured surface: (a,b) weld at 1.61 MPa; (c) pipe.

a quenched compression-molded plaque has a lifetime that is much greater than for the slow-cooled resin. The cooling rate of the former is 40°C/min and of the latter, 0.5°C/min. Figure 13 shows the notch opening vs. time of the pipe specimens with different notch directions and of the mold plaque specimens with different cooling rates. Figure 14 shows the anisotropy of the pipe by measuring thermal shrinkage of slices sectioned from pipe and then heated at 137°C for 15 min. These results are useful in understanding the difference between the lifetimes of the weld and of the pipe since they experience different cooling rates and different morphologies with respect to their molecular orientation.

4. DISCUSSION

When the molten end is removed from the hot plate, the surface begins to cool primarily by convection. An experiment was done to estimate how much the surface temperature changes during the 2 s dwell period. The experiment was done on a hot strip that was being extruded from a die at 220°C. It was found that in 1 s after hitting the air the temperature decreased by 35°C.[8] Considering that the molten zone has a temperature of 172°C before hitting the air and that the dwell time was 2 s, it is estimated that immediately prior to butting the interface the skin temperature is about 130°C. However, the temperature of the molten zone just below the surface quickly approaches 172°C. When the pressure is applied, the remnant skin does not flow out of the interface as rapidly as does the adjacent liquid, which is at a higher temperature because it has a lower viscosity. However, the higher the pressure, the better the removal of the cooler skin. Once the cooler skin is removed, the mixing and diffusion of the molecules readily occurs in the interface, and thus good adhesion occurs between the two pipes.

The region of poorest adhesion is likely to occur in the center of the pipe wall. As shown in Figure 4(b), the melt near the outside surface flows outward to form the outside bead and the melt near the inner surface flows inward to form the inside bead. Toward the center of the wall, the flow rate tends to go to zero and forms a dead zone. Thus, the remnant skin that acts as a barrier to adhesion is likely to be found in the center of the pipe wall, as pointed out by Atkinson et al.[1-3] in Figure 2.

If a contaminant also occurs at the surface, it will be more effectively removed by the higher pressure. The work by Aoki et al.[4] shows that a very high pressure will completely remove the remnant skin, but will also produce a high degree of molecular orientation, which lowers the resistance of the weld to slow crack growth.

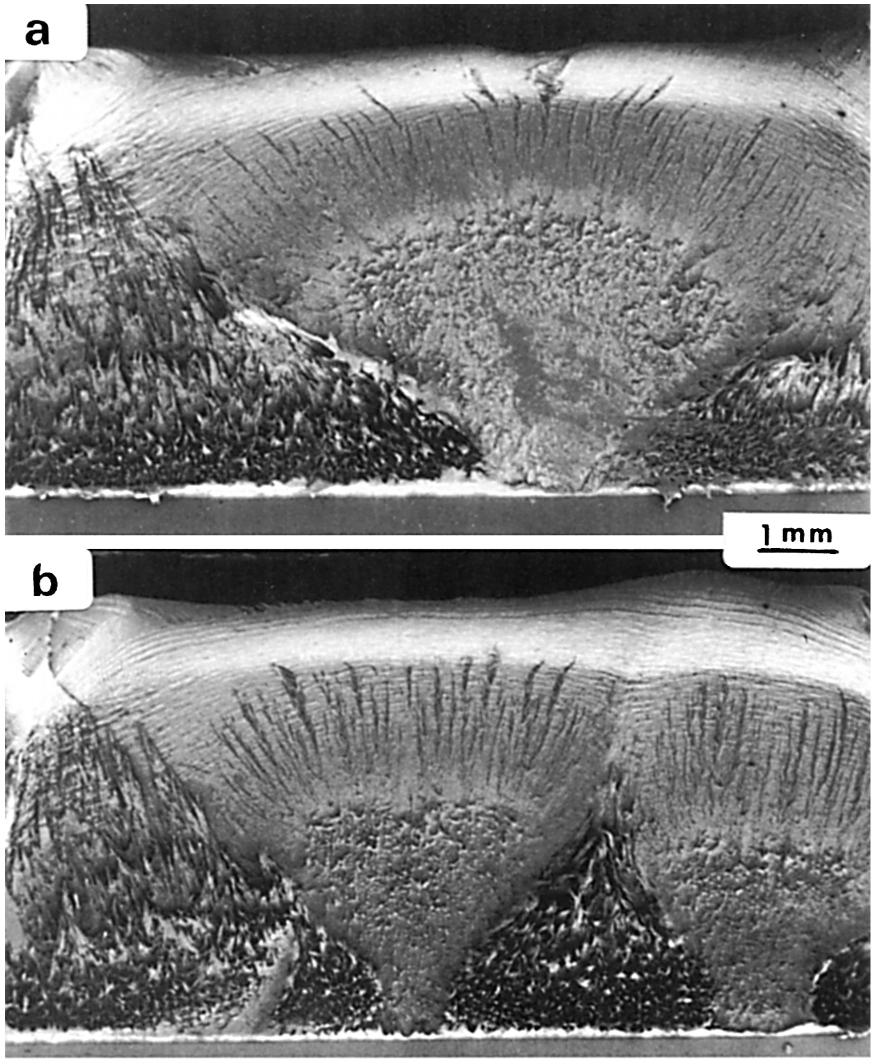

Figure 9 Examples of unbonded areas at weld pressure of 0.31 MPa.

It is interesting that the Vaseline and graphite spray did not affect the lifetime. Presumably, the flow rate under the high pressure was sufficient to remove both of these contaminants along with the remnant skin. Teflon is a very bad contaminant for the obvious reason that its melting point is 260°C

Table III Effect of Anisotropy and Thermal History on Lifetime

Notch Direction or Thermal History	Lifetime (min) (80°C, 2.4 MPa)	Average
Notch ∥ z axis[a]	1041, 900	971
Notch ⊥ z axis[a]	1557, 1397	1427
Quenched plaque	1616, 1580, 1233, 1012	1360
Slow cooling molded plaque	176, 150, 181, 268	194

[a] Sample from the pipe. Notch was made on inside surface of the pipe.

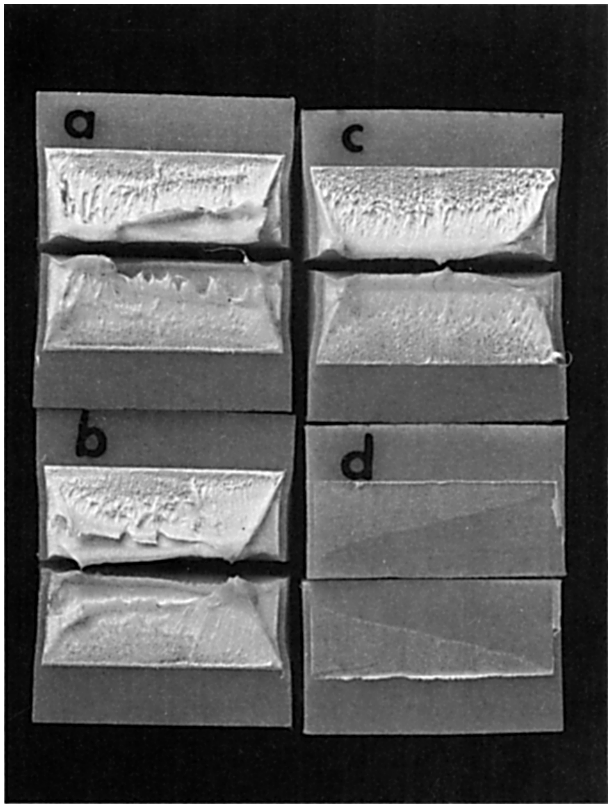

Figure 10 Fractured surface of weld joints contaminated by (a) Vaseline, (b) graphite, (c) and (d) Teflon. Weld pressure is 1.61 MPa for (a–c), and 0.31 MPa for (d).

compared to about 140°C for polyethylene. Thus, the Teflon coating never melted during the welding operation and acted as a barrier to the diffusion process that is necessary to cause adherence at the interface. The high pressure probably removed most of the remnant skin that contained Teflon, but at low pressure, the Teflon remains at the interface.

It is generally observed that the resistance to slow crack growth of the butt weld is approximately one-half that of the pipe, as shown in Table I. This difference is caused primarily by two factors, assuming that the remnant skin has been completely removed by imposing a sufficiently high pressure: The pipe and the weld have a different degree and direction of molecular orientation and they were exposed to different cooling rates. When the notch is parallel to the direction of orientation, the slow crack growth resistance is markedly decreased, as shown in Table III. The effect of notch orientation relative to the molecular orientation was measured in the case of the pipe, but only the case of the notch being parallel to the direction of molecular orientation in the butt weld could be measured. Table III also shows the profound effect of cooling rate where the faster the cooling rate the greater the resistance to slow crack growth. Preliminary analysis of the cooling rate of the pipe as compared to that of the butt weld indicates that the butt weld is cooled more slowly. These

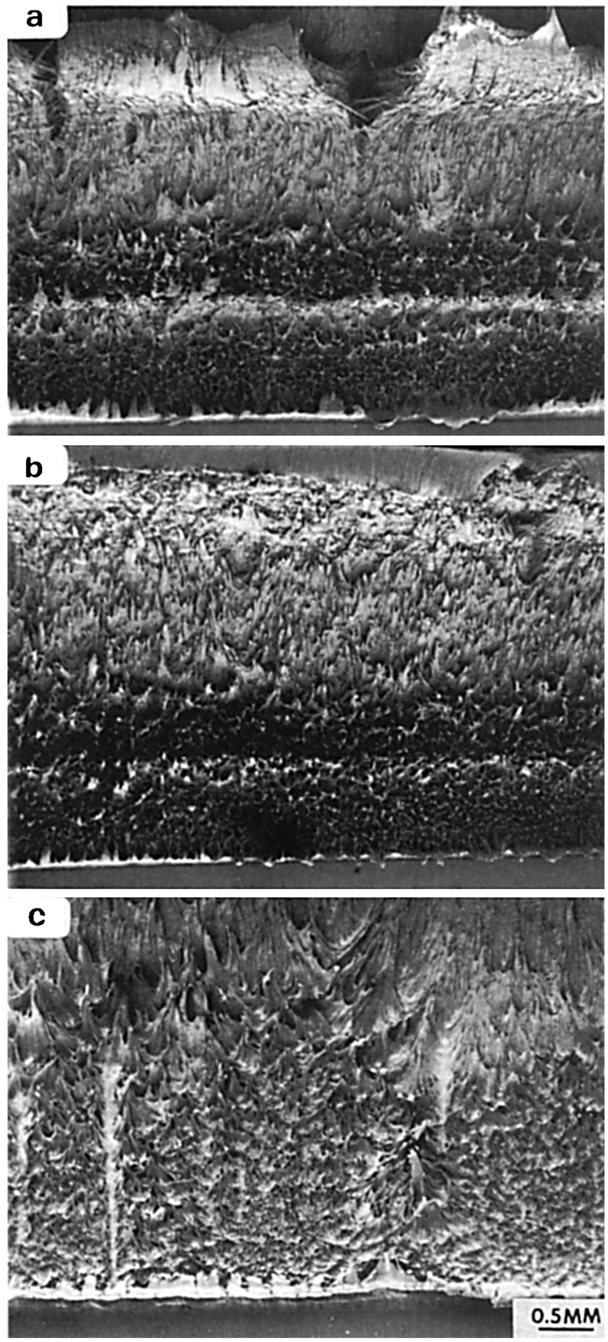

Figure 11 Fibrils in fractured weld contaminated by (a) Vaseline, (b) graphite, and (c) Teflon at pressure of 1.61 MPa.

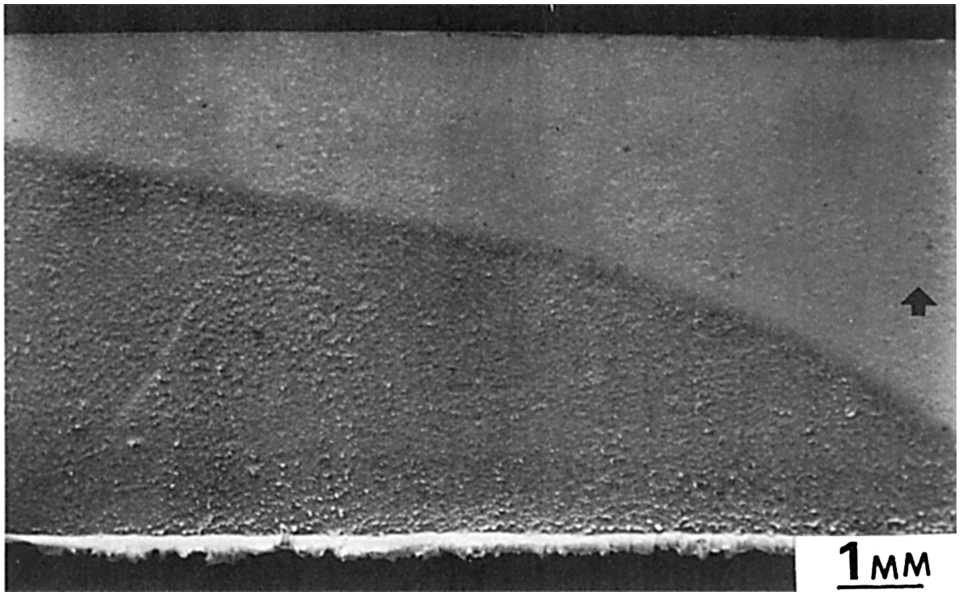

Figure 12 Fractured surface of weld contaminated by Teflon and welded at 0.31 MPa. Arrow shows direction of crack growth.

results strongly suggest that a difference in morphology between the pipe and the weld caused by a difference in molecular orientation and cooling rate can readily account for the fact that the pipe is twice

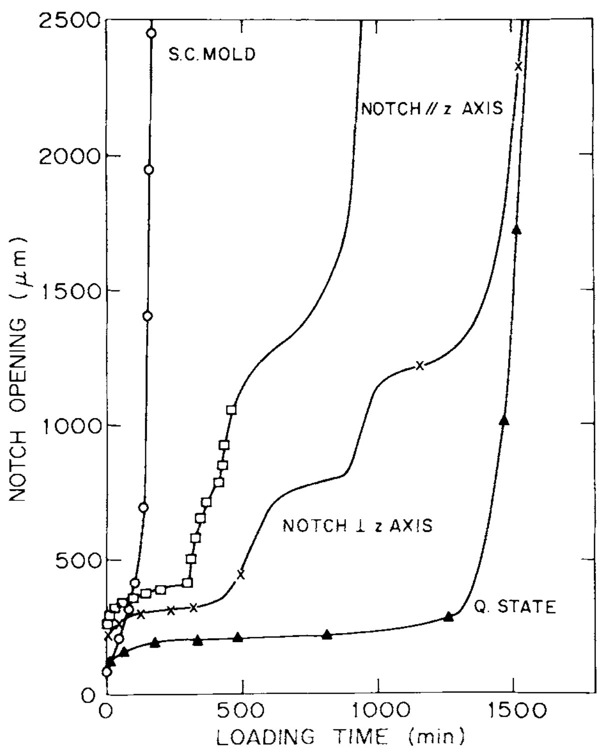

Figure 13 Notch opening vs. time for pipe specimens with notch in different directions and for isotropic specimens with different thermal history, tested at 80°C and 2.4 MPa.

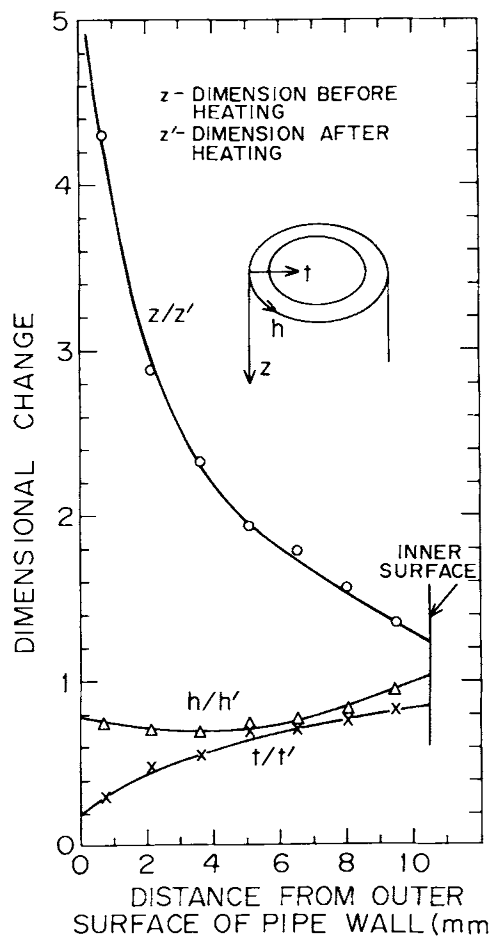

Figure 14 Shrinkage at 137°C after 15 min of thin slices sectioned from 110 mm pipe. z, t, and h express the axial, radial, and hoop directions of the pipe.

as strong as the weld with respect to slow crack growth resistance. More detailed experiments are required to more specifically account for the effect.

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REFERENCES

1. P. Barber and J. R. Atkinson, *J. Mater. Sci.*, **9**, 1456 (1974).
2. D. R. DeCourcy and J. R. Atkinson, *J. Mater. Sci.*, **12**, 1535 (1977).
3. J. R. Atkinson and D. R. DeCourcy, *Plast. Rubber Process. Appl.*, **1**, 287 (1981).
4. T. Aoki, M. Yasu, K. Simakawa, Y. Goto, N. Ishikawa, and T. Kamiya, in *Eleventh Plastic Fuel Gas Pipe Symposium Proceedings,* Oct. 1989, San Francisco, CA, p. 756.
5. S. M. Pimputkar, in *Tenth Plastic Fuel Gas Pipe Symposium Proceedings,* Oct. 1987, New Orleans, LA, p. 38.
6. X. Lu and N. Brown, *J. Mater. Sci.,* **26,** 612 (1991).
7. X. Lu and N. Brown, *J. Mater. Sci.,* **26,** 917 (1991).
8. X. Lu and N. Brown, unpublished data.

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