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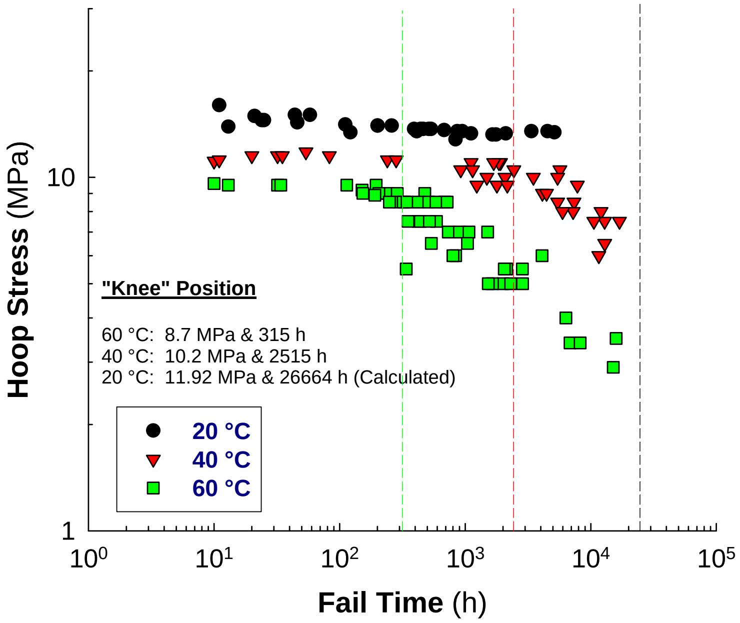
BACKGROUND

Polyethylene pipes are used extensively for the transportation and distribution of natural gas, with over 80% of the new piping installations using polyethylene. A majority of the field failures in polyethylene pressure pipe applications are attributable to a brittle slow crack growth (SCG) fracture that is characterized by the stable growth of a crack with little macroscopic plastic deformation. In an effort to understand the relationship between molecular architecture and pressure pipe SCG fracture, various lab-scale tests have been developed and optimized to capture the salient aspects of the field fracture. The Pennsylvania Edge-Notch Tensile Test (PENT; ASTM F1473) is one such outcome.

In the PENT test, a rectangular specimen that is notched on one side is subjected to creep. In other words, one end of the rectangular specimen is fixed in position while a load (dead weight) is hung from the other end. The primary purpose of the notch is to introduce a triaxial stress state at the notch tip to ensure a brittle SCG fracture mode. The load is chosen such that the stress at the notch tip (ligament stress = 2.4 MPa) is well below the yield stress of the material. The outcome of this test is the time to failure or the time it takes for the two halves of the specimen to fully separate or reach a separation of 0.5 inches, whichever comes first. . The resistance to SCG fracture is then related to the PENT failure time.

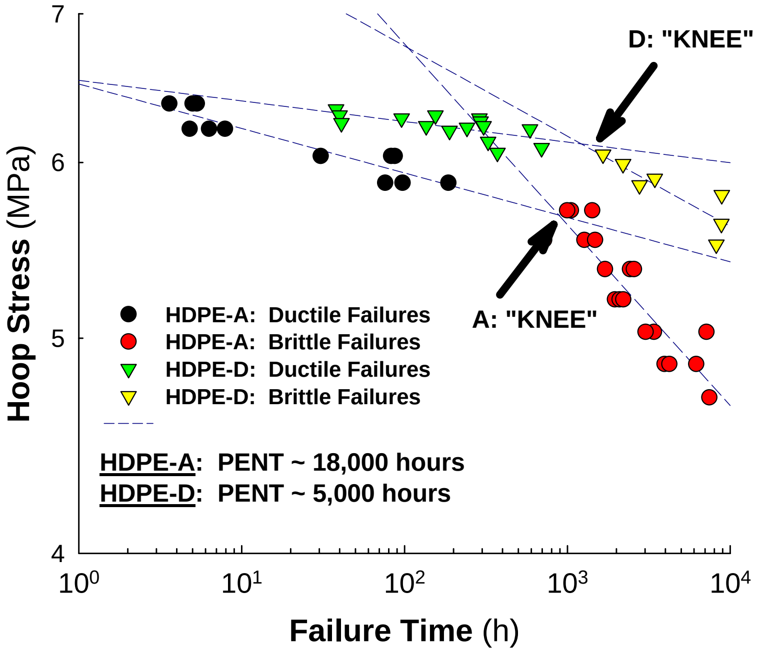
The design stress and the useful service lifetime of polyethylene pipes are typically estimated by performing creep rupture tests at multiple temperatures. In this test, the pipe of interest is subjected to a certain hydrostatic pressure (expressed as hoop stress) and the failure time is recorded; failure is defined as a continuous loss of pressure within the pipe. Typically, a log-log plot of pipe hoop stress versus failure time is constructed and extrapolated to a desired lifetime. Figure 1 is a representative plot of hoop stress versus failure time (at 20, 40 and 60 °C) for a given PE pipe. First, at all temperatures, we see a systematic increase in the failure time with decreasing hoop stress. We also note acceleration of the fracture process at higher test temperatures. The transition from ductile to brittle failure (knee) is clearly evident at the higher test temperatures (40 & 60 °C), while it is absent at 20 °C (within the testing period). The location of this knee shifts to shorter times and lower stress levels at higher temperatures. Based on the location of the knee at 40 °C and at 60 °C, the location of the knee at 20 °C is predicted using time-temperature superposition principles. In the ISO 9080 protocol, ductile and brittle failure data from the various test temperatures are combined using time-temperature superposition principles (the end-use temperature is the reference temperature) and the combined data are then extrapolated to a 50 year lifetime to determine the design stress of the pipe.



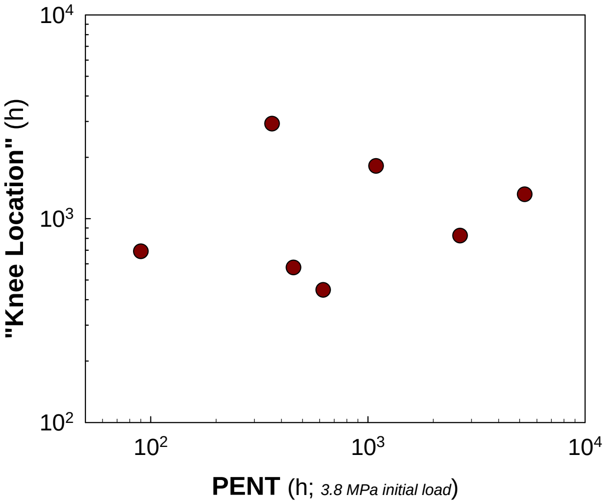
In this report, we seek to correlate the brittle fracture of pressurized polyethylene pipes (from creep rupture testing) to the PENT failure times obtained from compression molded specimens. A wide spectrum of polyethylene pipes were considered for this effort including those polymerized using chrome-oxide, Ziegler-Natta and metallocene catalyst technologies. Creep rupture testing was performed on nominal two-inch SDR11 pipes in accordance to ASTM D1598. All the subject pipes contained approximately 2.5 weight percent carbon black. PENT was performed in accordance to ASTM F1473 using compression molded specimens of the base polymers.

RESULTS AND DISCUSSION

In Figure 2, creep rupture data at 80 °C for two PE4710 pressure rated pipes (HDPE-A and HDPE-D) are shown (1). For both pipes, the "knee" or the transition from ductile failures at high stresses to brittle failures at low stresses is clearly evident. Specifically, the knee for HDPE-D occurs at much longer times compared to that of HDPE-A. Further, for any given stress within the brittle fracture regime, the failure time for HDPE-D is considerably longer. These observations clearly indicate that the HDPE-D pipe is generally more resistant to SCG fracture compared to the HDPE-A pipe. However, the PENT failure times for the two polymers indicate otherwise with HDPE-A displaying much longer failure times (~18,000 hours) compared to HDPE-D (~5,000 hours).

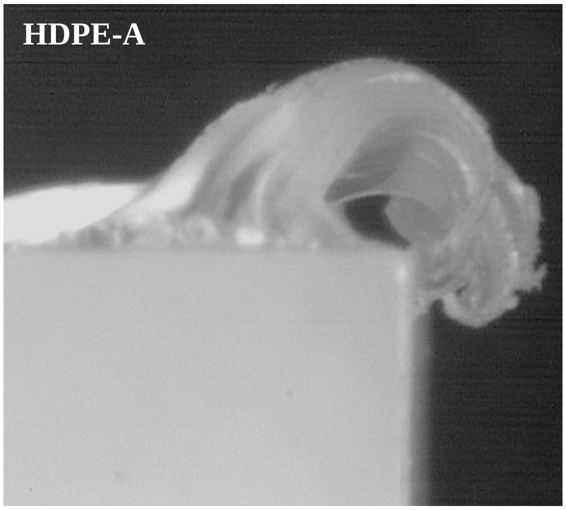


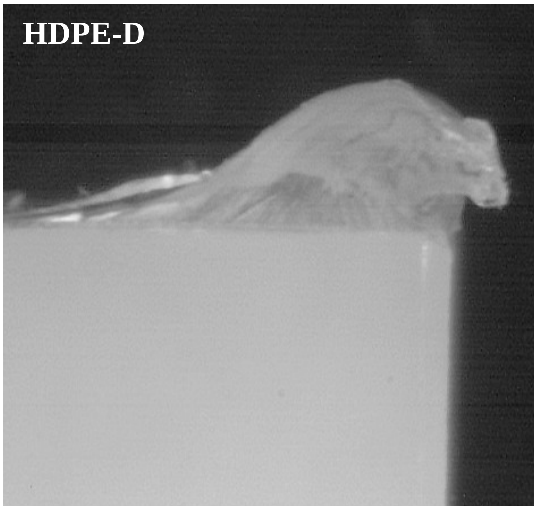
The PENT time to failure is often used to rank and grade various polyethylenes in terms of their SCG resistance. In Figure 3, the location (time) of the knee at 80 °C for various polyethylene pressure-rated PE pipes is plotted as a function of their PENT failure times (Note that the PENT data in Figure 3 was obtained at a higher stress of 3.8 MPa initial load rather than the standard 2.4 MPa as required in the ASTM F1473 PENT standard. This was done to accelerate the PENT failure times for several of the higher performance resins in this study, which as the data in Figure 3 confirms approached several thousand hours even at these higher initial load conditions). It is clear from this figure that the PENT failure times are inadequate to predict the location of the knee in polyethylene pipe creep rupture testing. Therefore, one has to be extremely cautious in relying on the PENT test to predict the SCG performance of the ensuing pipe.



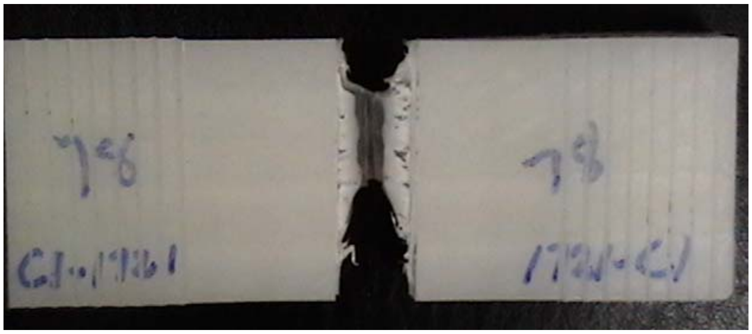
While we now recognize that the PENT measurement does not have much value in terms of predicting the occurrence of brittle failures in pipe creep rupture testing, let us try to examine the dynamics of the PENT test in some more detail. During the PENT test, when a notched specimen is subject to a constant load, many stages of deformation are usually prevalent. However, only the ultimate failure time is recorded and reported. As a first approximation, the failure time can be considered to be composed of three sequential deformation regimes; they are: (a) Fracture Initiation Step; (b) Slow Crack Growth; and (c) Post-Yield Tensile Stretching. The fracture initiation step is the time involved in the creation of the craze fibrils that initiate the fracture process. Once the craze fibrils have developed, the crack propagation is accomplished through the rupture of the extended craze fibrils. Many investigations indicate that the fracture process in a creep test (like PENT) occurs in a step-wise fashion with the craze-zone formation and crack growth processes proceeding sequentially (2-5). As the crack propagates, the ligament area decreases; this means, the ligament stress increases steadily during the test. It is then clear that the ligament stress will eventually exceed the yield stress of the polymer at some point during the test such that notch-blunting occurs and arrests the crack growth process. Subsequent deformation of the specimen occurs in a ductile fashion with ultimate failure occurring after the fibrils (in the ligament) connecting the two halves of the specimen have been stretched completely.

Figure 4 shows the side-views of failed PENT specimens for HDPE-A and HDPE-D. The edge opposite to the notched edge is shown here. These images clearly show macroscopic yielding for both polymers, with the extent of post-yield tensile stretching being considerably greater for HDPE-A, although, as mentioned previously, the test time would have been stopped when the total separation reached 0.5 inches. While it is clear that post-yield tensile stretching occurs towards the end of the PENT test, the relative contribution made by this deformation to the ultimate failure time is unclear.





In Figure 5, the stretched portion of the PENT specimen is shown; this picture was taken on a specimen after the test was stopped prior to ultimate failure. This is another clear indication that post-yield tensile stretching (ductile fracture) exerts a considerable influence on the PENT failure time.



The PENT failure times encompass the time-scale for fracture initiation, crack propagation and post-yield tensile stretching. The brittle fracture of pressurized polyethylene pipes does not include a macroscopic ductile component. Therefore, it is perhaps not surprising that there is no correlation between PENT and brittle fracture of pressurized polyethylene pipes.

CONCLUSIONS

In this investigation, a wide spectrum of polyethylene resins was chosen such that they differed considerably in their architectural and compositional make-up. These polymers were converted into pipe (constant dimensions) under fairly similar extrusion processing conditions. These pipes were subjected to extensive creep rupture testing (hydrostatic pressure testing) at multiple hoop stress levels and temperatures. In our analysis of pipe creep rupture fracture, we found that the high-stress PENT (measured at 3.8 MPa initial stress as opposed to the standard 2.4 MPa initial stress prescribed in the governing ASTM Standard F1473) failure times did not correlate well with the brittle failure onset times in pressurized pipes. . We do not believe that this discrepancy and lack of correlation between the two tests is related to our use of the higher initial stress condition, although that remains a possibility. Rather, we believe that this discrepancy may be related to the fact that the current ASTM F1473 standard, while certainly recognizing and calling attention to the issue of “extensive deformation” (i.e., ductile failure mode) during the PENT test lifetime, does not provide a precise way of dealing with the transition of brittle to ductile failure mode. In other words, while the PENT test reports a (total) time to failure, with failure defined as “Failure occurs when the two halves of the specimen separate completely or extensive deformation occurs in the remaining ligament”, it implicitly assumes that this total PENT failure time is all in the brittle failure mode. We are unsure, given our work and data presented here, if that assumption holds true for all PE resins, particularly the newer, higher performance PE4710 type resins. If the underlying polymer architecture causes the relative contributions of brittle and ductile failure times within the PENT (total) failure time to vary, that could explain why the PENT failure time does not correlate well with the pipe brittle failure onset time (6). We believe this issue of relative brittle and ductile failure modes (and respective failure times) within the lifetime of the PENT test need to be further investigated. Since the PENT failure time may be viewed to be a composite of the time period required for fracture initiation, crack propagation and post-yield (ductile) tensile stretching, while the brittle fracture of pipes subjected to creep rupture testing does not include a macroscopic ductile fracture component per se, it seems conceivable that such a situation may confound the correlation of these two tests. We are continuing to explore this issue to understand it better. However, given our findings to date, we remain cautious in interpreting the true value of the PENT test when developing polymers and pipes for high-performance pressure pipe applications. Lastly, we believe that the pipe industry at large would be well served to look into the PENT test in more depth in order to resolve some of the seeming, and significant, discrepancies that arise out of our work.

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(6) Unpublished CPChem data - In ongoing work since the time of the original report, we have found, for example, that molecular weight appears to have a significant influence of the total PENT failure time. One resin of extremely high M_w (~ 500,000 g/mol) and broad MWD (M_w/M_n ~ 70) yielded a standard (2.4 MPa) PENT failure time > 12,703 hours, at which time it was taken off test. This resin exhibited pressure pipe brittle failure onset at 80 °C of only 825 hours. Conversely, another experimental resin, with much lower M_w (~250,000 g/mol) and narrower MWD (M_w/M_n ~ 20), but higher levels of short chain branching in the high M_w fraction, exhibited a standard PENT failure time of 2,016 hours while exhibiting a pressure pipe brittle failure onset at 80 °C of 2923 hours.