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플라스틱 파이프의 잔류응력에 관한 연구
IV. 아닐링에 따른 변형 및 파괴성질의 변화

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Residual Stresses in Plastic Pipes and Fittings
IV. Effect of Annealing on Deformation and Fracture Properties

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요 약: 잔류응력 상태의 폴리에틸렌 파이프를 아닐링하여 이에 따른 변형 및 파괴거동을 고찰하였다. 일반적으로 아닐링은 잔류응력을 완화 또는 제거함과 동시에 폴리에틸렌의 밀도를 증가시키므로 이들 각각이 큰 변형 및 파괴 거동에 미치는 영향을 split 링 인장, 단시간 파이프 파열, 환경응력균열, 긴 시간 파이프 압력-파괴 및 정적균열 파괴실험을 통해 분리하였다. 잔류응력의 완화는 아닐링 시간보다, 아닐링 온도에 더욱 민감하였고, 120 ℃에서 1시간의 아닐링은 파이프내의 잔류응력을 완전히 제거하였다. 아닐링은 큰 변형 성질인 인장강도 및 단시간 파열강도를 아닐링전과 비해 각각 약 20% 증가시켰고, 파괴성질인 긴 시간 압력-파괴 강도와 정적균열 성장속도를 각각 약 100% 증가시켰다. 이 결과들로부터 큰 변형거동은 밀도, 그리고 파괴거동은 잔류응력상태에 의존함을 알 수 있었고, 환경응력균열 실험은 큰 변형이 가해진 상태에서 진행되는데 이로 인해 잔류응력이 완화 또는 제거되므로 잔류응력에 의한 파괴거동을 가리는데 있어 효과적인 방법이 될 수 없었다.

ABSTRACT: The effect of thermal annealing on deformation and fracture behavior of residually stressed polyethylene pipes was investigated. Thermal annealing of the pipes simultaneously caused a change in material density and the relaxation of residual stresses. The residual stress relaxation was more sensitive to annealing temperature than annealing time. Annealing at 120 ℃ for 1 hour removed the residual stresses effectively. While the large deformation properties such as the split ring tensile yield strength and the pipe burst strength were affected primarily by the change of crystallinity, the fracture related properties were predominantly affected by the residual stress relaxation. Sustained long term strength and slow crack growth were such properties. It was demonstrated that both of these properties were sensitive to residual stress change and that 100% increase in fracture times was observed as a result of tensile residual stress relaxation. Although the environmental stress cracking resistance (ESCR) test was also conducted to measure the relative cracking behavior the test was not suitable for determining the residual stress effect, as large deformation applied on ring sample effectively removed the residual stresses.

Keywords: plastic pipes, residual stress and distribution, annealing, large deformation behavior, fracture behavior.

INTRODUCTION

The long-term behavior of polyethylene pipe materials is dependent on several factors, including the residual stress. It was demonstrated that the residual stresses and their distributions exist through the wall thickness of the polyethylene pipes.[1-6] Also, it was found that a maximum biaxial tensile residual stress is present at the inner surface and a maximum biaxial compressive stress at the outer surface. Thus, it is expected that the mechanical behavior will be affected. In general, the study of the residual stress effect involves a procedure of thermal annealing, which attempts to systematically vary the residual stress states. The problem with thermal annealing is that not only there is a change of residual stress states, but also creates material changes such as density and detailed crystallite morphology. As a consequence, the mechanical properties are influenced and thus affecting the structure performace of the pipes. Hence, the property change due to annealing could be attributed to either a density change or a residual stress change or both. For polymers it is generally shown that the residual stresses will influence solvent stress cracking resistance, brittle fracture strength, fatigue strength and impact strength,[7-11] although no such studies exist specifically with regard to plastic pipes.

In the application of low and medium pressure rated polyethylene pipes and fittings, the tensile yield strength, quick burst strength, sustained long-term strength, ESCR and the slow crack growth characteristics of the pipe resins basically determine their performances. The first two are the large deformation related properties, while the latter three are the fracture properties. In the presence of residual stresses these properties are affected, but to which degree is not yet reported for the polyethylene pipes. The magnitude of density change and residual stress relaxation upon annealing has been determined by the authors.[1] However, how each of the change affects deformation and fracture behavior has not been reported. The importance of such is that it will allow the proper choice of experiments to be performed in rating the plastic pipe materials, having the residual stress effect accounted for. In this paper, results relating the annealing effects to tensile yield strength, quick burst strength, long-term strength, ESCR and slow crack growth in residually stressed polyethylene pipes are presented. The results are summarized with regard to separating the effect of residual stress relaxation and the morphological changes to pipe mechanical properties. For this, various tests were performed on as-received and annealed pipes and correlations were made between tests to demonstrate each effect of material density change and residual stress relaxation on deformation and fracture properties. Also, a finding on ESCR test method is included in the context of its validity for measuring the residual stress effect.

EXPERIMENTAL

Material. The material used for the investigation was the medium density PE2306IA polyethylene pipe grade resin.[12] The material was received in as-extruded pipe form, having 2SDR11 size (i.e. 50 mm nominal diameter and standard dimensional ratio (SDR)-outside diameter to pipe wall thickness ratio of 11). To achieve systematic variation in magnitude and distribution of the residual stress state along the pipe wall thickness, some of the pipes were annealed at 80 ℃, 100 ℃ and 120 ℃, for 1 hour, 10 hours and 100 hours. Density changes upon annealing were determined by a density gradient column per ASTM D-1515.

Residual Stress Measurements. The residual stresses in both the annealed and as-received pipes were determined by using turning and boring methods, and ring slitting methods, presented in detail in the Part I of this paper series.[13] Same procedures described in Part 1 were followed for the sample preparation. The ring samples utilized for the annealed pipes were sectioned from pipes following thermal annealing. The maximum longitudinal and circumferential residual stresses at the outer and inner most pipe surfaces were approximated by the slitting techniques and Equations 1 and 2, respectively.[2]

$$\sigma_{H(max)} = \pm \frac{E(t) \times h_0}{1-\nu} \frac{D_2(t) - D_1}{D_2(t)\, D_1} \quad (1)$$

$$\sigma_{L(max)} = \frac{E(t) \times h_0 \Delta(t)}{L^2} \quad (2)$$

Here, $E(t)$, h_0, ν, $\Delta(t)$, L are the creep modulus, wall thickness, Poisson's ratio, tongue end deflection, and tongue length, respectively. D_1 and $D_2(t)$ are the mean diameters of the ring sample before and after slitting. The circumferential residual stress distributions through the wall thickness were obtained using boring and turning methods and Equation 3.[13]

$$\sigma\theta(h) = \pm \frac{E(t)}{12\pi \phi^2} \Big\{ 4h\delta(h) + h^2\delta'(h) + 2\int_0^h \delta(h)dh - \frac{1}{h_0}\Big(4\int_0^{h_0} h\delta(h)dh + \int_0^{h_0} h^2\delta'(h)dh + 2\int_0^{h_0}\int_0^h \delta(h)dhdh\Big)\Big\} \quad (3)$$

Here, δ is the gap displacement and δ' is the differential δ with respect to the current wall thickness. The plus and minus signs preceeding the right hand side represent turning and boring method, respectively. Also, for turning and boring ϕ becomes the inside and outside ring diameters, a and b, respectively.

Equation 3 is evaluated by using $\delta(h)$ and $\delta'(h)$, which are the quantities experimentally determined.

Split Ring and Film Tensile Tests. Tensile tests were carried out on split ring specimens obtained from both annealed and as-received PE2306IA pipes. Split ring samples were produced by first drilling 6.35 mm diameter holes, centered at every 13 mm along the pipe length, completely through the pipe diameter. Rings were then removed from the pipe by cutting at each hole center, and milled to a 12.5 mm height. The schematic diagram of the split ring sample containing a reduced section is shown in Fig. 1. The split rings were tensile tested in an Instron machine, using the split ring tension fixture (Fig. 1). This fixture was designed in such a way that it allowed to have rings tensile tested without altering the geometry or introducing additional stresses during testing. A 5.0 mm/minute cross-head speed was used to obtain load-elongation curves. Film samples of 0.130 mm thickness were also prepared by remolding pipes, using a compression molding. The films were either water quenched (4 ℃), or air cooled. Some films were then annealed at 120 ℃ for one hour. ASTM sized tensile film samples were produced in a Dewes

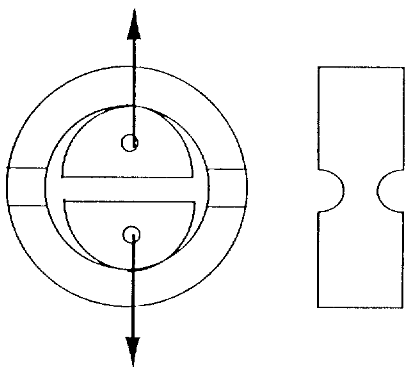

Figure 1. Schematic diagram of split ring tensile specimen and loading geometry.

Gumbs Die Expulsion Press. The load-elongation curves were determined by using the cross-head rate of 5.0 mm/min.

Quick Burst and Sustained Internal Pressure Testing of Pipes. Quick burst test and sustained internal pressure test on annealed and as-received pipes were conducted per ASTM D1599 and ASTM D1598 methods, respectively. In the quick burst test, the end capped pipes were pressurized in such way that the time to failure was achieved within 60 to 70 seconds. The corresponding burst pressures were recorded. The sustained internal pressure tests were carried out at 80 ℃ and by applying a constant internal pressure that resulted in a hoop stress value of 4 MPa on2SDR11 pipes. Failure times corresponding to the constant applied pressure were recorded. For 80 ℃, 100 ℃ and 120 ℃ annealed pipes, 1 hour anneal time was applied prior to quick burst and sustained pressure tests. Pipe samples were approximately 305 mm in length and end caps (Plexco-2SDR11) were put on at each ends using butt fusion welding (McElroy Fusion Welder). For both quick burst and sustained pressure tests the same equipment was used and the details of the experimental setup is illustrated in Fig. 2. The setup basically consisted of an air driven water pump, an accumulator and a manifold containing submanifolds, each acting as a test station. Each station was equipped with a bypass valve and a velocity fuse for initial pressure application and quick shut off at specimen failure, respectively. The hoop stress on the pipe sample was produced by filling the pipe with water and subsequent pressurization at the test station. The internal pressure regulation and the failure time detect on were achieved by using pressure transducers interfaced to PC. The sudden drop in pressure reading was taken as the sample failure time. Test temperature during sustained pressure test was controlled to ± 0.5 ℃ accuracy, using distilled water as the heating medium. Some of the sustained pressure failed pipes were further analyzed for residual stresses by turning and boring methods.

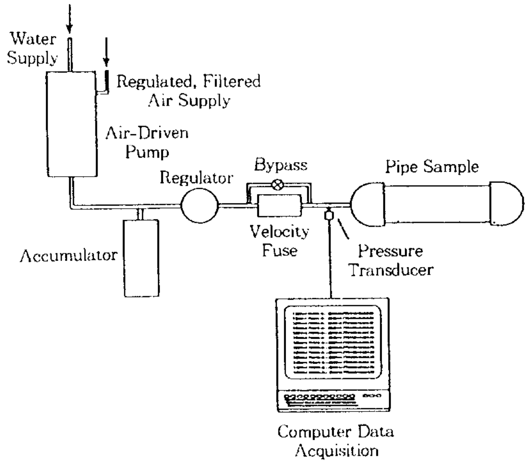

Figure 2. Test setup for pipe quick burst and internal pressure tests.

Compressed Ring ESCR Test. For the determination of ESCR times in as-received and annealed pipes, rings machined from 2SDR11 pipes (25 mm width) were utilized. A notch of 25 mm long and 0.5 mm deep was produced along the outer surface, in the circumferential direction (Fig. 3), by using a razor blade. Several rings were then placed in a test fixture and compressed until the top and bottom inner ring surfaces were in contact. The rings were placed so that the center of the notch length was positioned in perpendicular to the compression direction. The compression fixture-ring sample assembly was then placed in a beaker containing 25% Igepal solution and tested at 50 ℃, and the ESCR times were recorded. The ESCR time was defined as the time at which compressed ring samples displayed a crack growth perpendicular to the razor notch.

Slow Crack Growth Measurements. The slow crack growth behavior in as-received and annealed pipes were investigated by using the ring SCG sample described in Part Ⅲ of this

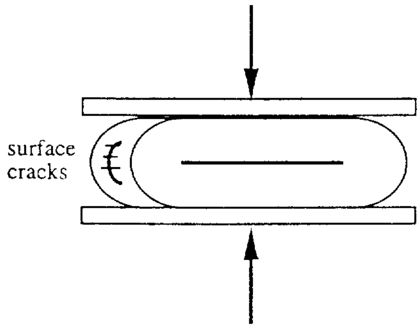

Figure 3. ESCR compression ring test sample and loading geometry.

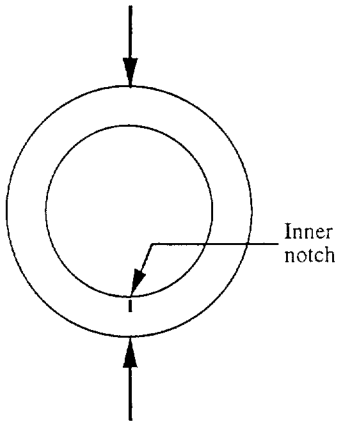

Figure 4. SCG ring specimen.

paper series.[14] For this investigation, 25 mm wide rings obtained from the pipes were notched at the inner surface to a depth of 0.5 mm. The notch location with respect to diametrical compression loading axis is shown in Fig. 4. The slow crack growth experiments were performed under a constant diametrical compression (constant force of 445 N) in 25% Igepal-75% deionized water solution at a temperature of 50 ℃. The slow crack growth measurements were achieved by monitoring the crack extension with time. For this, slow cracked samples were taken out at intervals of load duration time up to where there was a substantial crack growth through the sample wall thickness. The ring samples were then washed in a distilled water to remove Igepal and impact fractured in a liquid nitrogen to reveal the crack surfaces. The crack length at each load duration time was obtained by measuring the crack length at several different locations of the crack front, using a traveling microscope (30X).

RESULTS AND DISCUSSION

When residually stressed polyethylene pipes are thermally annealed to remove the residual stresses, not only is there a residual stress relaxation but also creates material changes such as density and detailed crystallite morphology. Both of these changes in turn are expected to affect the mechanical behavior of pipes and thus their structural performances. Hence, a better understanding of how the residual stress and the density change alter tensile yield strength, quick burst strength, sustained long-term strength, ESCR and slow crack growth behavior of the pipe, can provide the basis for designing better performing plastic pipe resins.

The change of maximum tensile residual stresses in the PE2306IA 2SDR11 pipe, determined by the slitting method, is shown in Fig. 5 with respect to annealing temperature and time. Similarly, the change of wall thickness residual stress distribution with annealing temperature and time is illustrated in Fig. 6. Also, density changes are tabulated in Table 1 for various annealing temperatures and times. These results clearly indicate the simultaneous change in the residual stress state and density brought about as a result of thermal annealing. The density change was minimum for the 80 ℃, and with 1 hour annealing no change was observed when compared to the as-received density. However, for the same annealing condition (80 ℃, 1 hour), a noticeable relaxation of residual stresses occurred such that about 30% reduction was observed for both maximum compressive and tensile residual stresses (Fig. 5). With 100 ℃ and

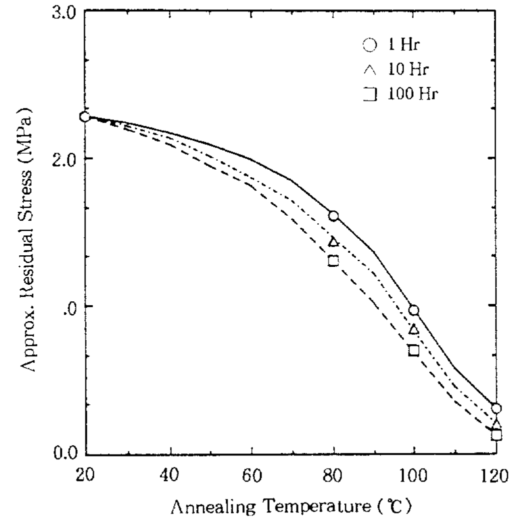

Figure 5. Change of residual stress with annealing in 2SDR11 PE2306IA pipes.

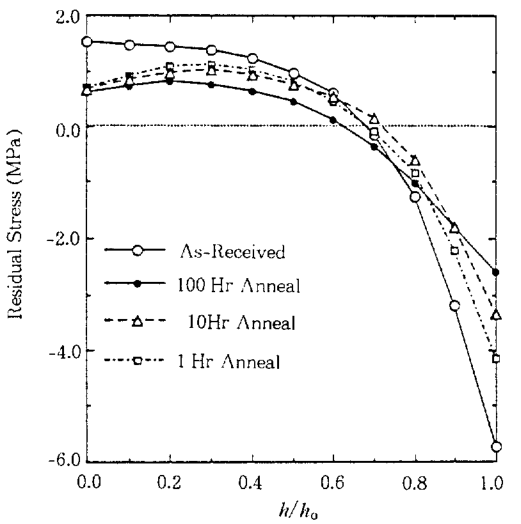

Figure 6. Change of residual stress distribution with annealing in 2SDR11 PE2306IA pipes.

120 ℃ annealing temperatures the density change became noticeable and for the latter, annealing time also played a significant role in increasing the density. In Fig. 5 it is interesting note that the relaxation of stresses were mostly affected by the annealing temperature and that the anneling time after the first hour played a minor role. Also, at

Table 1. Density Change upon Annealing for PE2306IA (Five Samples were Tested per Condition and the Standard Deviation was about ±0.0002)

anneal time (HR)	deinsity after annealing (g/cm^3)		
	80 ℃	100 ℃	120 ℃
1	0.9526	0.9545	0.9555
10	0.9533	0.9547	0.9565
100	0.9535	0.9548	0.9575
as-received density : 0.9526 g/cm^3			

120 ℃, the residual stress relaxation became almost complete. These results seemed to apply not only to PE2306IA 2SDR11 pipe but to all polyethylene pipe types and sizes tested as well.[1] Fig. 6 shows that the residual stress distribution was also similarly affected during annealing at 80 ℃, and while the tensile residual stress near the inner surface did not show much change with anneal time after the first hour, the compressive residual stress at the outer surface was more sensitive to the time of annealing.

Tensile Behavior. The effect of annealing on tensile yield strength of pipes, as measured by the split ring tensile specimen is tabulated in Table 2. The yield strength of the 80 ℃, 1 hour annealed pipe was not different from that of the as-received pipe. However, a steady increase was obtained with the higher annealing temperatures. For 120 ℃, 1 hour annealed pipes, approximately a 19% increase can be observed. To help determine whether this increase was due to annealing induced residual stress relaxation or the density change, tensile test results from compression molded thin film samples were examined. In the case of water quenched thin films (0.130 mm thick), no residual stresses were assumed to be present because no temperature gradient along the film thickness was created during cooling due to the thickness. Therefore, the property difference between water quenched and annealed films can be only accounted for by the density change.

Table 2. Variation of Tensile Yield Strength with Annealing Determined from PE2306IA-2SDR11 Pipe and Compressing Molded 0.130 mm Thick Film

specimen type	annealing conditions			
	as-received	80 ℃/1 Hr	100 ℃/1 Hr	120 ℃/1 Hr
ring tensile strength (MPa)	15.69	15.69	16.51	18.65
film strength (MPa)	15.44	–	–	18.71

In Table 2, the magnitude of yield strength change between water quenched and annealed films is similar to those between as received and 120 ℃ annealed pipes. The stress-elongation curves of pipe and film samples are also shown in Fig. 7. It can be seen that the films closely simulated the properties of the as-received and annealed pipes. The similarity between the film and pipe tensile characteristics imply that the effect of annealing on tensile yield strength of pipe was due primarily to a change in crystallinity rather than the relaxation of residual stresses. It should be also noted that 80 ℃, 1 hour annealing did not change the density, however reduced the maximum residual stress by a measurable degree. The fact that the spit ring yield strength did not change, despite the measurable change of residual stress, is indicative of residual stress not being a factor for the tensile yield strength behavior.

Quick Burst Behavior. The results from the quick burst test for as-received and annealed pipes are illustrated in Fig. 8. In the quick burst test, the measured quantity was the internal pressure that gave rise to a ductile yielding failure within a given time frame, which was between 60 and 70 seconds. The burst pressure is seen to rise with annealing temperature and the magnitude of increase between the as-received and 120 ℃ annealed pipes is approximately equal to those of tensile yield strength measured from the split ring test (17% versus 19% increase, respectively). Thus, the increase in burst pressure with annealing can be similarly attributed to the increase in density rather than to residual stress relaxation.

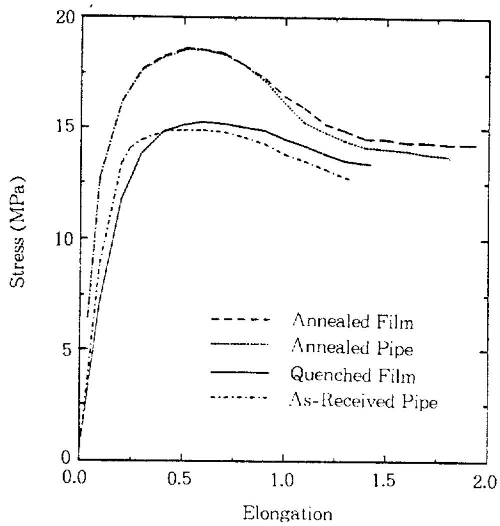

Figure 7. Effect of annealing on stress-elongation curves for ring and film samples.

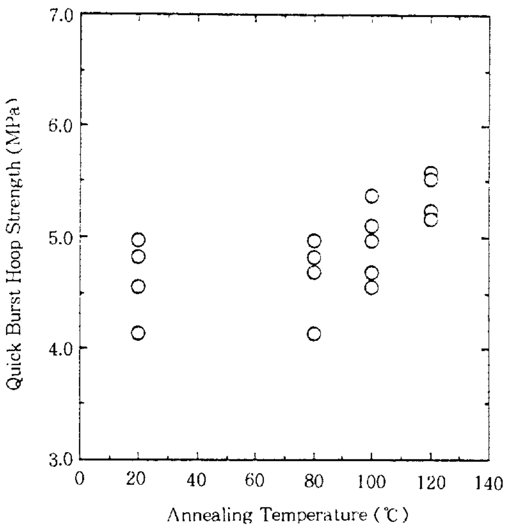

Figure 8. Effect of annealing on quick burst test of pipes.

ESCR Behavior. ESCR behavior of as-received and annealed pipes is shown in Fig. 9. There exist a trend demonstrating the improved ESCR performance for specimens annealed at 100 ℃ and above. The ESCR test was conducted

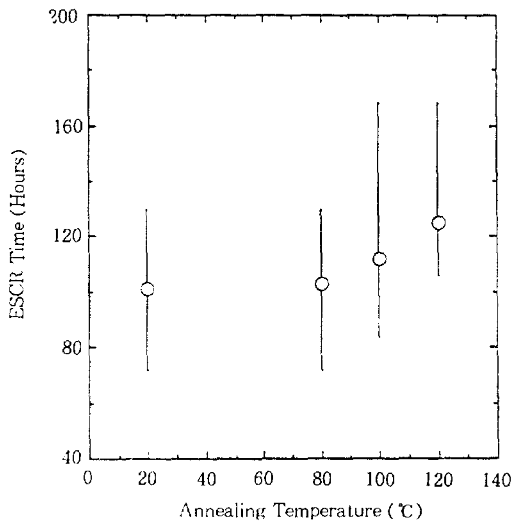

Figure 9. Effect of annealing on the ESCR performance.

in such a way that surface crackings were induced at the outer surface, hence the region of compressive residual stress. In view of this, the result shown in Fig. 9 is interesting because the compressive residual stress effect was not at all observed. That is, since the compressive residual stress is a maximum in the as-received pipe, the ESCR performance should been the best compared to annealed pipes. In the ESCR test, the rings were compressed until two opposite inner surfaces became in contact with each other. As a result, a large tensile plastic deformation was produced at the outer surface, where the environmental stress cracking was to develope. Thus, the compressive residual stresses at the outer surface are likely to be relaxed by the plastic deformation and hence making the ESCR test not suitable for testing the residual stress effect. In view of the report that the ESCR times were known to decrease with increasing crystallinity,[15] it is not clear whether the increase in the ESCR time for higher temperature annealed pipes can be attributed to the increased crystallinity. However, the fact that 80 ℃ annealing did not change the ESCR times and that the magnitude of change between as-received and 120 ℃ annealed pipes was similar to those observed in the tensile yield and quick burst strengths (20% vs. 19% vs. 17%, respectively) does point to a conjecture that the ESCR times were mainly affected by the density changes.

Sustained Pressure Behavior. The performance of the plastic pipes are primarily rated by the long-term sustained internal pressure testing.[16] In this test a constant internal pressure is applied until the pipe failure occurs. The magnitude of the sustained pressure applied is well below the pressure needed to cause delayed yielding failure of the pipe, however in the range to produce brittle (or slit) failures. Failure time is the quantity measured and this is detected by a leak in the pipe or a sudden drop in the internal pressure, as a consequence of the pipe failure. The failure time for the brittle failure mode is the sum of crack initiation and crack propagation times. Since the hoop stress developed as a result of internal pressure application is the highest at the pipe inner surface, and that a tensile residual stress exist at this surface, cracks are generally initiated at the inner pipe surface and propagate toward the outer surface. Hence the increased failure time implies that the crack initiation was either delayed and/or crack propagation rates became reduced. The results of annealing temperature versus failure time curve of pipes, tested at 80 ℃ and 4 MPa sustained hoop stress, are shown in Fig. 10. As the annealing temperature increased, a substantial increase in failure time occurred. For samples annealed at 80 ℃ and 120 ℃ for 1 hour, 40% and 100% increase can be respectively observed, which is far greater in magnitude than those property increase caused mainly by the density changes, as discussed before. In view of the fact that brittle (slit) fractures in pipe failures were known to accelerate with increasing crystallinity, that is lower failure times,[12] such a large in-

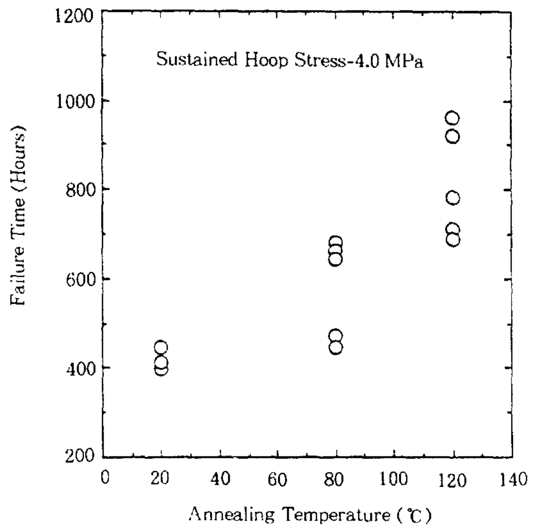

Figure 10. Effect of annealing on sustained pressure-failure time behavior.

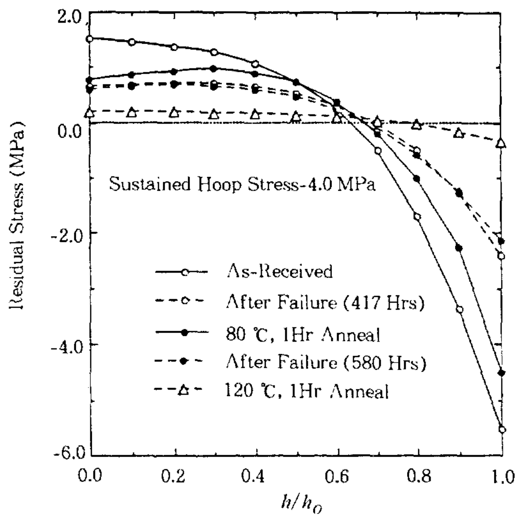

Figure 11. Residual stress distributions before and after sustained pressure testing.

crease in failure times with annealing can be attributed in major part to tensile residual stress relaxation at the inner pipe wall surface and not the density change. To more closely examine the difference in failure times between the as−received and the annealed pipes, the residual stresses were measured before and after the sustained pressure test. The results are shown in Fig. 11. For the as-received pipe, the mean failure time was 417 hours and there is a tensile residual stress relaxation similar to 80 ℃, 1 hour annealed pipe, although the compressive residual stress was further relaxed. In the case of 80 ℃, 1 hour annealed pipe, relaxation was smaller in magnitude with mean failure time recorded at 581 hours. Thus, when compared to Fig. 6 the sustained pressure had a minor effect on tensile stress relaxation at the inner surface. Pipes with 120 ℃, 1 hour annealing prior to testing, exhibited a mean failure time of 815 hours. For this pipe, the prior annealing effectively eliminated the residual stress in the pipe and hence the measurement after the test was not necessary. Approximately a 160 and 400 hour differences in failure times between as-received and 80 ℃ annealed pipes, and as-received and 120 ℃ pipes, respectively, indicate that the initial residual stress magnitude is important and do support the relaxation of tensile stresses at the pipe inner surface as the predominant mechanism by which to retards the crack initiation and or crack propagation processes.

Slow Crack Growth Behavior. The slow crack growth results obtained from the inner notched SCG ring specimens are shown in Fig. 12. Distinct differences in crack growth behavior can be observed between the cracks initiating and propagating from tensile (as-received) and zero (120 ℃, 1 hour annealed) residual stressd states at the crack tips. For example, to achieve a slow crack growth of 2.5 mm, it took about 100% less for the as-received specimen compared to the annealed specimen. This finding is significant because the magnitude of this difference in failure times was also observed during sustained pressure test between the as-received and the annealed pipes. As noted before, the failure time in the sustained pressure test is the sum of crack initiation and crack growth times and the failure begins at

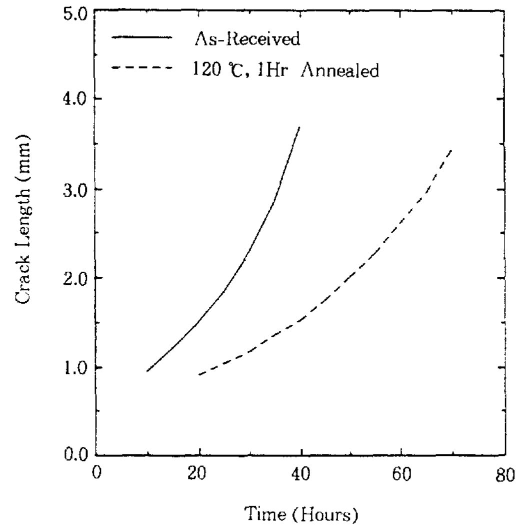

Figure 12. Slow crack growth behavior between inner notched annealed and as-received specimens.

the tensile residual stress region. Hence the improvement of the slow crack growth time for annealed specimens can be similarly attributed, in major part, to a relaxation of tensile residual stresses at the inner surface and not by the density change due to annealing. The physical evidence for the residual stress effect was shown in the Part Ⅲ of this paper in term of the crack front shape on the fracture surface.[14]

CONCLUSIONS

The effect of thermal annealing on deformation and fracture behavior of residually stressed polyethylene pipes was investigated. In general, thermal annealing induced a density change and simultaneously produced residual stress relaxation. While the large deformation properties such as the split ring tensile yield strength and the pipe burst strength were affected primarily by the change of crystallinity, the fracture related properties were predominantly affected by the residual stress relaxation. The sustained pressure and the slow crack growth were such properties. It was demonstrated that both of these properties were sensitive to residual stress change and that 100% increase in fracture times was observed as a result of tensile residual stress relaxation. Although ESCR test also measured the relative cracking behavior the test was not suitable for measuring the residual stress effect, as large deformation applied on the ring sample effectively removed the residual stresses.

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