

ASME Journal of Pressure Vessel Technology Online journal at: https://asmedigitalcollection.asme.org/pressurevesseltech



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Experimental Research on Thermal-Oxidative Aging Performance of Polyethylene Pipe Under Hydrostatic Pressure

Aging performance of polyethylene (PE) material from PE pipe under hydrostatic pressure was investigated by measuring its mechanical/chemical properties. Tensile test, thermogravimetric analysis (TGA), oxidation induction time (OIT), as well as hardness test were carried out to evaluate the aging status of polyethylene material. Results show that as aging time prolongs, elongation rate after brake, thermal decomposition temperature, as well as oxidation induction time of PE specimen decrease, while its hardness increases, indicating that its mechanical/chemical properties change with the aging intensity. Life prediction model was also established based on failure time–pressure data obtained from hydrostatic pressure test, under the guidance of Arrhenius theory. Conclusions drawn from this research could help to prompt the efficiency of PE pipe on-site inspection as well as serve as references for understanding the aging behavior of PE pipe. [DOI: 10.1115/1.4066448]

Keywords: polyethylene pipe, differential scanning calorimetry, thermogravimetric analysis, aging behavior, life prediction

1 Introduction

Easy to transport/deploy, being resistant to corrosion, these advantages have made polyethylene (PE) pipe a priority in civil gas pipeline industry; now more than 70% of newly deployed civil gas pipe are PE pipes in China. It has been more than 40 years since China deployed its first gas PE pipeline in 1980s, yet the recognized life span of PE pipe in transportation system is 50 years; aging problem of PE pipe has become a practical problem that must be faced. How to deal with these pipes that near the end of their life span sparked intense debate among engineers and scholars. Many researchers have focused on this problem but few of them could reach a consensus. Many factors lead to aging of PE pipe, heat, UV light, strong acids or bases environment, pressure, etc. Since gas PE pipe is typically buried underground, their ambient environment is assumed to be stable, and the transported medium, pressurized purified natural gas, contains little impurities. The main factor that leads to aging of PE pipes is stress load generated from transported medium.

The aging process of polyethylene is essentially the combination of free radicals and reactive oxygen species in its molecular chain, which results in an increase of oxygen content in its molecular chain. A series of phenomena caused by carbon–hydrogen main chain fracture initiated by oxidation, and it can trigger a sequence of reactions of molecular chains and ultimately lead to degradation and cross-linking [1]. Degradation reaction leads to a reduction of polyethylene molecular weight, which will result in the decrease of tensile strength and modulus. Cross-linking makes polyethylene harder and more brittle, resulting in the decrease of elongation rate after break [2]. The interaction between carbon-hydrogen chain and reactive oxygen in polyethylene is affected by two factors: one is the oxygen absorption capacity of the molecular chain. Some studies [3–6] have shown that hygrothermal environment could boost the diffusion rate of reactive oxygen in the molecular chain of polyethylene, thus accelerating the aging of polyethylene, and hygrothermal environment is the common external environment for gas polyethylene pipes in service; the other is the microsurface area of polyethylene materials. The microsurface area of polyethylene materials is directly related to surface microcracks, and the generation of surface microcracks is related to the creep degree of polyethylene materials under long-term dynamic loads [7]. The above two factors cause the aging of polyethylene pipes to occur first on the surface, making polyethylene becomes harder and more brittle, and more prone to produce microcracks, which allows the hygrothermal environment to diffuse into the inside of polyethylene. The above processes promote each other, making the polyethylene pipe material gradually age from outside to inside [8].

Wang et al. evaluated the aging damage of PE pipe thermal butt fusion welds using nonlinear ultrasonic technique as a nondestructive testing method. Ultrasonic harmonics are generated due to aging damage, and the acoustic nonlinearity parameter based on the fundamental and second harmonics is calculated [9]. Redhead et al. investigated the chemical resistance of PE for pipe applications with special regard to physical and chemical material aging. Potentially physical or chemical material aging was investigated by the degree of crystallization, the oxidation induction time (OIT), and via infrared-spectroscopy [10]. Bredács et al. investigated the impact of disinfected water on the degradation of PE with immersion tests of two PE pipe materials in 10 and 5 ppm chlorine dioxide

¹Corresponding author. Contributed by the Pressure Vessel and Piping Division of ASME for publication in the JOURNAL OF PRESSURE VESSEL TECHNOLOGY. Manuscript received March 12, 2024; final manuscript received August 28, 2024; published online September 21, 2024. Assoc. Editor: Julian Hallai.

(ClO₂) medium at 60 °C. Material aging at 50 °C and above was found to be much faster than at 40 °C applying 1 ppm ClO₂ concentration. An optimized testing condition for fast material characterization in case of 1 mm thick specimens was found to be a concentration of 1 ppm ClO₂ at 50 $^\circ C$ [11]. Lin et al. investigated the oxidation induction time, melt mass flow rate, carbon black content, and density of five gas polyethylene pipes with different service times; results show that the longer the pipe use time, the greater the change rate of oxidation induction time and melt mass flow rate, and the weaker the oxidation performance and flow performance of the pipe. They found out that the oxidation induction time and the melt mass flow rate are suitable for evaluating the aging performance of the pipe, while the carbon black content and density are not suitable for evaluating the aging performance of the pipe [12]. Ulloa et al. investigated the creep behavior of a high-density polyethylene before and after exposure to oil derivates using transient rhinometry; the creep behavior of the original high density polyethylene was described using a generalized Kelvin-Voigt model based on two retardation times [13]. Dai et al. quantitatively studied the influence of different factors on PE thermo-oxidation properties by calculating the correlation coefficient between different factors and PE bending strength; results showed that lower of density, higher degree of crystallinity, and wider molecular weight distribution of PE results in faster decrease of the bending strength which was mainly concentrated in the early and latter part of aging [14]. Dai et al. conducted artificial accelerated aging test on low-density polyethylene in 80 °C thermal-oxidative environment for different time periods up to 64 days; results showed that thermal stability and melting characteristic decreased due to chain scission and branching. The characteristics value of low-temperature melting peak decreased and chain scission was predominant when the shorter chain increased gradually after successive self-nucleation and annealing technology [15]. Lan et al. proposed a prediction model for PE pipes by using the dynamic curve linearization method based on experimental data; research showed that the tensile mechanical properties of PE gas pipes change significantly with the increase of thermal-oxidative aging temperature, internal pipe pressure and aging time, and the load at the breaking point drops dramatically [16]. Chen et al. studied thermal-oxidative aging behaviors of PE pipe under constant pressure and cyclic pressure in various temperatures; result shows that the fracture strength of PE pipe decreases as the aging time prolongs, and a faster decreasing rate is also observed under the condition of cyclic pressure, compared to that under constant pressure. They proposed a lifetime prediction models of PE 100 pipe under constant pressure and cyclic pressure [17]. This article is the only one that the author can retrieve that has discussed about PE pipe aging issue under pressure load.

Based on the long-term hydraulic test of PE pipe conducted at Zhejiang University, this paper investigated the aging performance of PE pipe under external load. Internal pressure was introduced to perform aging treatment upon PE pipe samples. Chemical/ mechanical properties test was conducted upon PE pipe samples with different degree of aging. Conclusions drawn from this research could help to prompt the efficiency of PE pipe on-site inspection as well as serve as references for understanding the aging behavior of PE pipe.

2 Experimental Processes

2.1 Polyethylene Specimen Preparation. Polyethylene specimen for mechanical/chemical test was obtained from PE pipe samples (De90SDR11, PE100) after long term hydrostatic test. Mechanical properties tests include [18]: (1) single axis tensile test to obtain its elongation rate after brake, test sample was selected as type 1B, according to ISO527-1: 2019, as shown in Fig. 1, and (2) hardness test to obtain its Brinell hardness (Fig. 2). As a nonmetallic material that possesses strong viscoelasticity, hardness test data of PE cannot represent its true Brinell hardness, but could represent the transition process of its properties from ductileness to brittleness during aging process. Chemical properties tests of PE specimen









Fig. 1 PE specimen for single axis tensile test



Fig. 2 Illustration of PE specimen hardness test



Fig. 3 NETZSCH 204 HP differential scanning calorimeter and test specimen

include [19]: (1) thermogravimetric analysis (TGA) to obtain its thermal decomposition temperature, and (2) differential scanning calorimetry (DSC) to obtain its oxidation induction time (Fig. 3). TGA test was conducted under argon atmosphere, initial heating rate $2.8 \,^{\circ}$ C/min, and will gradually accelerate over time, test temperature $40-1400 \,^{\circ}$ C. DSC test is to analyze the OIT of PE material under different aging time (0 h/200 h/500 h/1000 h), and test samples of PE material weighted 15–30 mg are heated from room temperature to melt under oxygen flow at 50 ml/min. System will automatically record the time from endothermic to exothermic of the samples.

2.2 Aging Treatment and Lifespan Prediction Model. In this research, long term hydrostatic test was conducted to perform aging treatment of PE pipe samples under the guidance of code ISO9080-2012 [20,21]. All test samples are picked from the same batch of product, their properties are assumed to be stable, and pipeline specification is De90SDR11, as shown in Fig. 4. Test platform is illustrated in Fig. 5. This platform contains six pressure channels that output value of each channel is adjustable individually. Water was chosen as pressure medium, and test chamber temperature was set at 65 °C constantly. Pipe samples were conditioned in water chamber over 24 h prior to test and were then connected to pressure pump via pressure hose. System will keep the pressure value constant until the pipe bursts and record the time-pressure value automatically. When test time exceeds 10,000 h, the hydrostatic test will be terminated, and the remaining pipe samples were marked as "test end, not failed sample," and the corresponding failure time was recorded as 10,000 h.

Polyethylene pipe lifespan prediction model under long-term hydrostatic pressure was established based on time temperature equivalence principle, that allows us to predict the viscoelastic properties of an amorphous polymer at one temperature from measurements made at other temperatures [21,22].

3 Test Results and Discussion

3.1 Mechanical Properties Test. Single axis tensile test was conducted on Zwick/Roell Test Xpert II universal testing machine,







Fig. 4 Configuration of test samples



Fig. 5 Illustration of hydrostatic test platform

and four set of test data were obtained from tensile specimen, as shown in Fig. 6. These specimens were obtained from PE pipe samples that were tested under hydrostatic pressure for 0 h, 200 h, 500 h, and 1000 h, and tensile speed was controlled at 20 mm/min.

The decrease in elongation after fracture increases the failure probability of PE pipes during natural disasters. For example, in cases of mudslides or landslides, newly deployed PE pipes can withstand greater erosion intensity or soil slip, whereas aged PE pipelines with reduced elongation rate after brake may be unable to bear larger slip volumes. In extreme cases, aged PE pipes can fail faster due to fracture compared to newly deployed PE pipe.

Elongation rate after brake for each specimen is shown in Fig. 7, and it can be observed that as hydrostatic test time increases, elongation rate of tensile test specimens is recorded decrease, indicating that with the increase of hydrostatic test time, the thermaloxidative decomposition reaction of polyethylene material molecules occurs, making the plasticity of polyethylene material decrease, and this results in a gradual embrittlement of tensile test



Fig. 6 Tensile test specimen under hydrostatic pressure (from up to down: test time 0 h, 200 h, 500 h, and 1000 h)



Fig. 7 Elongation rate after brake of PE specimen under different aging time

specimen cut from pipe samples. Compared to aging time 0 h test specimen, elongation rate after brake of aging time 1000 h test specimen decreased from 407.03% to 368.70%.

Tensile strength of PE specimens experienced the similar variation trend as elongation rate after brake, as can be found in Fig. 8, and tensile strength for aging time 0 h specimen is 88.9 MPa, while this value decreased 11.58% to 78.6 MPa for aging time 1000 h specimen, indicating that aging treatment could also affect the tensile strength of PE materials. This conclusion lays the foundation for the following PE pipe life span prediction process.

Hardness test of PE specimen was also carried out here. In order to qualitatively illustrate the impact of aging treatment upon PE material, two specimens are selected for hardness test, one is heated to melt state and then cooled to room temperature in the air, as shown in Fig. 9. The aging level of this artificial aging specimen is assumed to be 100%. The other specimen is from newly produced PE pipe that its aging level is assumed to be 0% [23] (Fig. 10).

Test results are shown in Fig. 11, as can be observed that the Brinell hardness of new specimen is between 105 HB and 131 HB, while this value for artificial aging specimen has exceeded 360 HB. Ignoring the random error in the test, the Brinell hardness of artificial



Fig. 8 Strain-stress curve of PE tensile test



Fig. 9 Artificial aging specimen



Fig. 10 Hardness test surface of two specimens

aging specimen is about three times of that of new specimen. The aging process of polyethylene is essentially the combination of free radicals and reactive oxygen species in its molecular chain, which results in an increase of oxygen content in its molecular chain, and it can trigger a sequence of reactions of molecular chains and ultimately lead to cross-linking, making polyethylene harder and more brittle [24].

3.2 Chemical Properties Test. Differential scanning calorimetry test and TGA test are carried out to analyze the chemical



Artificial aging specimen



New specimen

Fig. 11 Brinell hardness test of two kinds of specimens



Fig. 12 DCS curves of different specimens

properties of PE material during aging process. DSC test is to analyze the OIT of PE material under different aging time (0 h/200 h/ 500 h/1000 h). OIT serves as an index to indicate the anti-oxidant consumption of PE materials, and shorter OIT means less antioxidant residues in the PE material, indicating a higher aging degree of the material. Test specimen is shown in Fig. 3, and DSC curves of different specimens were shown in Fig. 12. OIT of samples under different aging treatment time is shown in Table 1, and the corresponding test time–OIT variation trend is illustrated in Fig. 13. It can be observed from those figures that under the condition of constant temperature and constant pressure, with the increase of pressure loading time, the isothermal OIT of polyethylene pipe shows a trend of shortening with first-order exponential power, and the decreasing rate of pressure loading time in the 0–500 h period is greater than that in the 500–1000 h period, indicating that the

Table 1 OIT of samples under different aging treatment time

Test sample No.	Aging treatment time (h)	OIT (min)	
1	0	53.4	
2	200	46.7	
3	500	38.0	
4	1000	35.8	



Fig. 13 OIT trends under different loading time

thermal oxygen aging reaction of polyethylene pipe material is more intense in the early stage, and the aging phenomenon is more obvious. This can be explained that in the early stage of aging the polyethylene material molecules undergo thermal oxygen reaction with oxygen in the high-temperature environment, which leads to the decrease of structural regularity of the pipe material and the increase of branched chains. However, in the later stage, the oxidation on the surface of the material gradually tends to be saturated, and oxygen cannot continue to penetrate, leading to a slowdown in the aging effect.

Thermogravimetric analysis aims to acquire the thermal decomposition temperature (Ts) of PE materials, so as to analyze the variation trend of thermal stability of PE samples under different pressure loading time [25]. TGA test was conducted under argon atmosphere, initial heating rate 2.8 °C/min, and will gradually accelerate over time, test temperature 40–1400 °C. Ts curves of PE specimens under different loading time are shown in Fig. 14, and the corresponding Ts data are listed in Table 2.



Fig. 14 Ts curves of PE under different loading time (pipe samples aging test condition: $T = 65 \degree$ C, P = 1.1 MPa, water bath condition)

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Table 2 Thermal decomposition temperature (Ts) of PE materials

Specimen No.	Test condition	Loading time (h)	Ts (°C)
1	65 °C, 1.1 MPa water bath condition	0	529.15
2		200	512.17
3		500	502.00
4		1000	486.83

It can be observed that with the increase of pressure loading time, Ts curve of polyethylene material shares the similar variation trend with isothermal OIT of polyethylene material, also showing a firstorder exponential power change trend. Experimental results show that under the combined effect of temperature and internal pressure, the polyethylene pipe material undergoes a thermo-oxidative degradation reaction, which leads to a decrease in thermal stability. Combining the test results of DSC and TGA, it is anticipated that the isothermal OIT and Ts results can be used simultaneously to characterize the aging performance of polyethylene materials [26]. The longer the isothermal OIT and the higher the Ts, the lower the aging degree of polyethylene materials and the healthier the pipeline operating state. Selecting 1000 h of experimental data (TGA) as example, and take the derivative of thermogravimetric data (d/dt) to obtain the thermogravimetric change rate of polyethylene material at different temperatures (differential thermogravimetric analysis (DTG)), as shown in Fig. 15. It can be seen that the point with the largest thermogravimetric change rate also corresponds to the point with the largest thermogravimetric change, and the presence of only one peak on the DTG curve implies that only the anti-oxidant has been consumed during the heating process, while the material itself remains undecomposed.

Section 3.1 has demonstrated qualitatively that the aging degree of PE material has some bearing to its hardness. Based on the above analysis, it is proposed to correlate the hardness measurements taken at various parts along the pipeline with the OIT of the pipe material. The hardness measurements serve as the independent variable, while the oxidation induction time will be the dependent variable. By analyzing the variation trend in both variables, a corresponding relationship between the degree of aging of the pipe material and its hardness can be established. The selected test materials are obtained from the inner pipe wall, outer pipe wall, cross section of the pipe wall (base material), the bulging area after long-term hydrostatic testing, and an artificially aged sample. Importantly, all these test materials are taken from the same sample pipe (five test samples in total), as illustrated in Fig. 16.

Tested samples are shown in Fig. 17, and the corresponding DSC curves are demonstrated in Fig. 18. It can be observed that OIT of five samples exhibits considerable deviation, and OIT for materials







Fig. 16 DSC test samples location



Fig. 17 Five samples after test

from bulging area is 17.1 min while this value is 19.8 min/22.4 min/ 37.3 min for material from inner pipe wall/outer pipe wall/cross section of the pipe wall. And OIT for artificially aged sample is too short to be calculated. In general, the higher the aging degree of polyethylene materials, the shorter the isothermal OIT. Taking their Brinell hardness, as shown in Fig. 19, and it can be found that the hardness values of each sample vary. Hardness for base material/ inner pipe wall/outer pipe wall/bulging area is 145 HB/220 HB/318 HB/405 HB. Overall, the hardness of the bulging area material is the highest, and the hardness of the pipe wall cross section material (base material) is the lowest. However, the hardness value of the artificially aged sample (365 HB) with the highest degree of aging is lower than that of the bulging area, indicating that the fully aged polyethylene material has become brittle and lost its bearing capacity. In general, within the normal aging range, the hardness of polyethylene material increases as the degree of aging increases.

In order to investigate the relation between PE material OIT and its hardness, the oxidation induction time of the artificially aged samples was arbitrarily set to 0, and the hardness values and oxidation induction time of each sample were combined into a dataset to obtain the variation trend in the hardness of polyethylene samples with the aging degree, as shown in Fig. 20. It can be seen that there is a positive correlation between the hardness of polyethylene material, the higher the aging degree of the polyethylene sample, the higher its hardness. This phenomenon can be used for on-site inspection of gas polyethylene pipelines. By testing the hardness of the pipeline surface and comparing it with that of the new pipeline, the aging degree of the pipeline can be qualitatively judged.

As can be found in Fig. 20, except for the situation where polyethylene becomes brittle due to the high degree of carbonization after complete aging, resulting in a decrease in hardness, the hardness of polyethylene pipe increases with the increase of aging







Fig. 19 Brinell hardness test of five samples

degree before complete aging. To characterize the aging degree of pipeline through hardness, we selected the outer wall hardness of the gas polyethylene pipeline with a 30-year of operation time as an indicator, assuming that the aging degree of polyethylene pipeline gradually deepens within 30 years without brittle phenomenon caused by complete carbonization. By comparing the outer wall hardness of polyethylene pipeline sample with an operation time less than 30 years with that of the above 30-year pipe, the aging degree of the pipeline sample can be characterized by the ratio between them.

A case study was carried out here to illustrate the relation between pipe aging degree and its pipe wall hardness. Multiple hardness measurements were taken on the outer wall of polyethylene pipelines with different operation time and averaged to obtain the variation trend in wall hardness with operation time. As shown in Fig. 21, after eliminating random errors through multiple measurements, the wall hardness value gradually increases with the increase of pipeline operation time. Average hardness value for pipe with operation time of 7 yr is 274 HB, while this value for pipe with operation time of 28 yr has increased to 320 HB. The service



Fig. 20 Hardness–OIT relation of five PE samples



Fig. 21 Hardness variation of PE pipe with different operation time

environment and parameters of these pipeline samples are not consistent, which leads to different internal and external environments causing pipeline aging. This indicates that after eliminating the differences in environmental factors causing pipeline aging, the pipeline aging degree is positively correlated with its hardness.

4 Service Life Prediction

Long-term hydraulic test was conducted, and 43 set of data (failure time-pipe wall hoop stress) are eventually identified eligible for further regression analysis; a three-parameter model was introduced here to fit the dataset after logarithmic treatment

$$\log_{10}\sigma = c_1 + c_2 \frac{1}{T} + c_4 \frac{\log_{10} t}{T} - e \tag{1}$$

where $c_1/c_2/c_3$ are constant, T denotes test temperature, and e denotes error variance.

Hoop stress–failure time relation is shown in Fig. 22. It can be observed that there are two branches within the curve that is divided by a knee, indicating the transition of PE pipe failure mode from ductile failure mode to brittle failure mode. Calculate both branches' sample mean and standard deviation separately, and their allowable error is obtained as 0.009487542/0.020638199. By taking one-sided confidence as 97.5%, the lower confidence limit is presented: 0.762852412/0.685455315.

Substitute both branches into Eq. (1) separately and fit the dataset with least square method fitting. Since long-term hydrostatic (LTH) test temperature is set as $65 \,^{\circ}$ C, the fitted equation can be simplified as



Fig. 22 Hoop stress-failure time curve after logarithm treatment

Table 3 Calculation of error variance of two branches

Index factor	Branch 1	Branch 2
Sample size	32	11
Sample mean	0.772339954	0.706093515
Sample standard deviation	0.022784158	0.025989074
Average sampling error	0.004027708	0.007836001
Confidence	0.975	0.975
Degree of freedom	31	10
<i>t</i> -distribution two-sided quantiles	2.355568282	2.633766916
Error variance	0.009487542	0.020638199

$$\log_{10}\sigma = -0.0792\log_{10}t + 1.046 - e_1 \tag{2}$$

$$\log_{10}\sigma = -0.385\log_{10}t + 2.1988 - e_2 \tag{3}$$

Equations (2) and (3) represent the simple one-sided lower 97.5% error band with 95% confidence intervals, and error variance, e_1/e_2 , is calculated as Table 3.

Equations (2) and (3) are obtained as

$$\log_{10}\sigma = -0.0792\log_{10}t + 1.0365\tag{4}$$

$$\log_{10}\sigma = -0.385\log_{10}t + 2.1782\tag{5}$$

According to ISO 9080: 2012, extrapolation time limit is dependent on LTH test temperature, and extrapolation factor, k_e , is the function of ΔT , where ΔT is defined as

$$\Delta T = T_t - T \tag{6}$$

 T_t is LTH test temperature, and *T* is expected service temperature of PE pipe. In this case, PE pipe LTH test temperature is 65 °C (338.15 K), and PE pipe expected service temperature is assumed to be 30 °C (303.15 K); ΔT is 35.

For extrapolation time, t_e , it is the function of the maximum test duration t_{max}

$$t_e = k_e t_{\max} \tag{7}$$

Here, t_{max} is determined by the mean value of top five specimen test duration under the same test temperature. In the case studied here, this value is calculated as 3.9272 after logarithmic treatment. According to ISO 9080: 2012, $k_e = 30$, t_e is obtained as

$$t_e = 3.9272 + \log_{10} 30 = 5.4043 \tag{8}$$

Thus, extrapolation time limit curve as well as original LTH test curve is presented in Fig. 23.

And extrapolation data under LTH test temperature of $65 \,^{\circ}$ C are shown in Table 4.



Fig. 23 Extrapolation time limit with one knee point

Table 4 Extrapolation time limit at 65 °C

T (° C)	ΔT	k _e	t_e (h)	t_e (a)
30	35	30	253,700	28.9

Table 5	Knee point at 65 °C	
		_

Temperature (°C)	Stress (MPa)	Time (h)	
65	5.31	8456	

The corresponding knee point is shown in Table 5.

As can be seen in Table 4, this set of PE pipe is estimated to serve 253,700 h (28.9 yr) before fail under 30 °C with hoop stress of 5.31 MPa, based on LTH test data under 65 °C. Material brittle–ductile transition point occurs at 8456 h that PE pipe failure mode has transformed from ductile failure mode to brittle failure mode at this point. This prediction is credible. The expected lifespan of PE pipes is 50 years. However, China deployed its first gas PE pipes since the 1980s, which is less than 50 years ago. No one has ever seen a truly aged PE pipe literally. Nevertheless, for safety reasons, engineers will consider replacing them when their service life approaches this value (around 30 years or 40 years). The above prediction is based on the lower confidence limit of test data, and the result is relatively conservative. Overall, the predicted lifespan of the PE pipe is generally consistent with the actual maintenance strategy for PE pipes.

5 Conclusion Remarks

The main purpose of this paper is to propose a rapid method for on-site inspection of PE pipe aging degree. Hydraulic test was introduced to perform aging treatment of PE pipe samples, and the mechanical as well as chemical properties of PE pipe samples with different aging time were investigated. Some conclusions can be drawn as below:

- (1) Differential scanning calorimetry and TGA tests were conducted on pipe samples with different aging time, and test results show that under the combined effect of temperature and internal pressure, PE pipe material undergoes a thermo-oxidative degradation reaction. Combining the test results of DSC and TGA tests, it is anticipated that the isothermal OIT and Ts results can be used simultaneously to characterize the aging performance of polyethylene materials.
- (2) Five test samples were selected to perform mechanical test to investigate the relationship between the degree of aging of the pipe material and its hardness; results show that there is

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positive correlation between the mechanical properties of PE material and its aging degree, within the normal aging range, the hardness of polyethylene material increases as the degree of aging increases.

- (3) Case study also reveals that the pipe wall hardness value gradually increases with the increase of pipeline operation time, indicating after eliminating the differences in environmental factors causing pipeline aging; the pipeline aging degree is positively correlated with its hardness.
- (4) Finally, service life prediction was performed, and results show that material brittle–ductile transition point was found to occur at 8456 h, and PE pipe service life was expected to be 28.9 yr at $30 \,^{\circ}\text{C}$.

Future study includes quantitative assessment of PE aging status based on combined on-site test data and lab test data, and establishment of improved life prediction model based on multitemperature test data, that could give out more accurate life prediction result and automatically identify data knee (PE material brittle–ductile transition point).

Acknowledgment

Many thanks to Ningbo OPR to provide convenient for conducting the test.

Funding Data

• Science and Technology Program of the State Administration for Market Regulation (2023MK054, China State Administration for Market Regulation).

Data Availability Statement

Data provided by a third party listed in the Acknowledgment.

Nomenclature

- DSC = differential scanning calorimetry
- DTG = differential thermogravimetric analysis
- OIT = oxidation induction time
- PE = polyethylene
- TGA = thermogravimetric analysis
- Ts = thermal decomposition temperature

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